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Rheological Analysis of Silicone Pavement Joint Sealants

by   Larry N. Lynch

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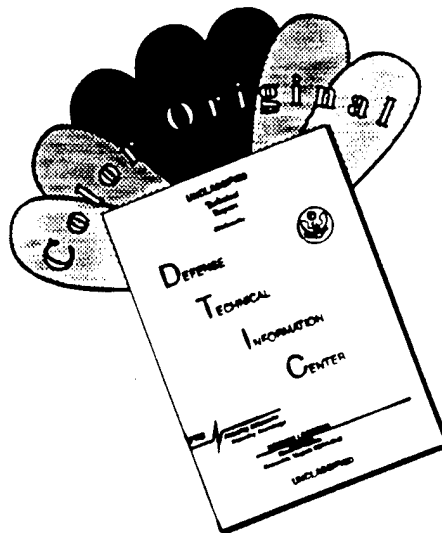
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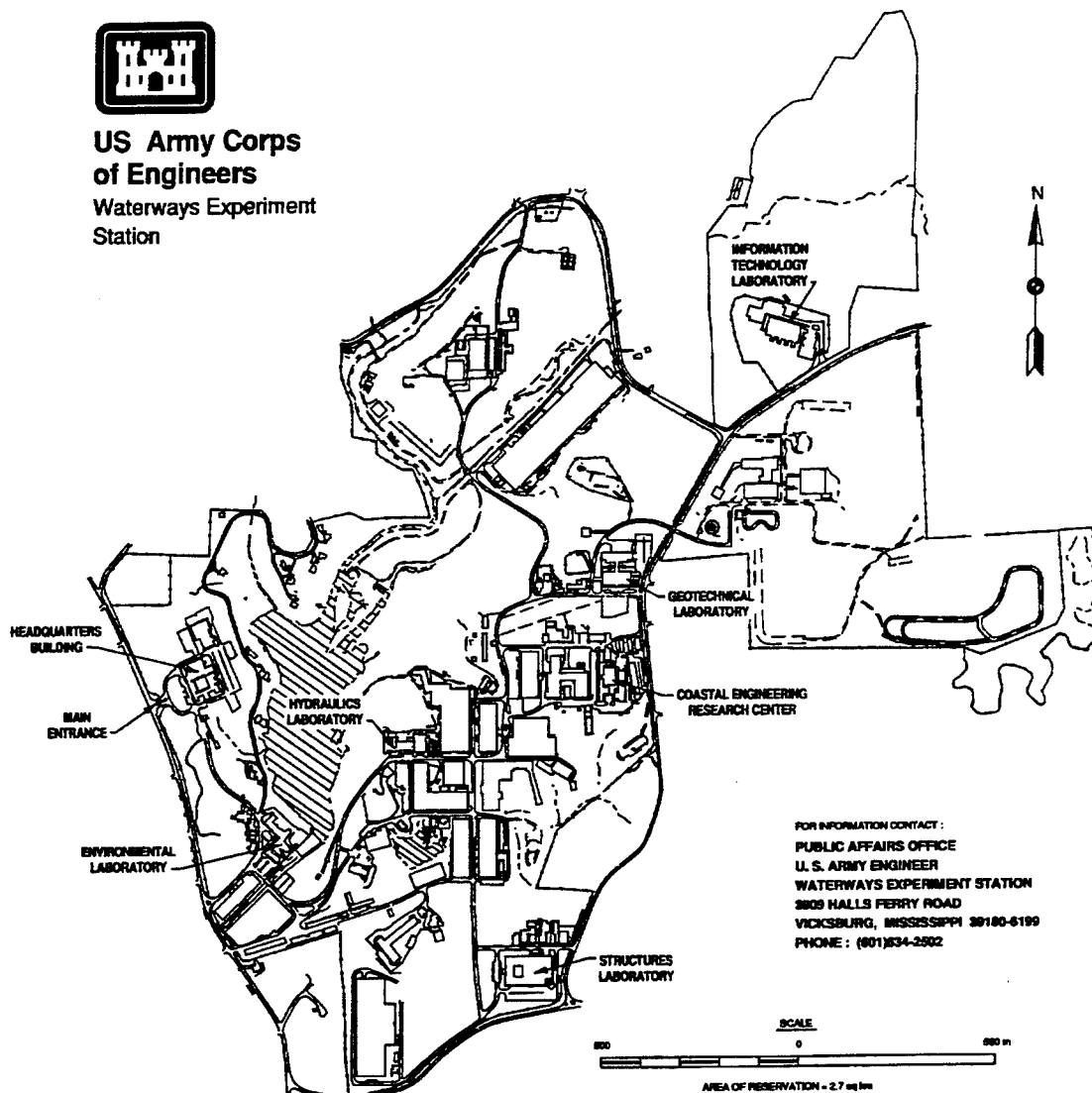3909 Halls Ferry Road
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Final report

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SCALE

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TABLE OF CONTENTS

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF TABLES (continued)

PREFACE

This project was conducted by the U.S. Army Engineer Waterways Experiment Station (WES), Vicksburg, MS, and was sponsored through the Laboratory Discretionary Research Program at WES. This report is a reprint of a Dissertation submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy to the University of Washington (UW), Seattle, Washington, August 1995. The Dissertation Supervisory Committee from the UW consisted of Dr. D. J. Janssen, Chairman, Drs. J. P. Mahoney, and G. S. Rutherford, Civil Engineering Department, and Dr. W. D. Scott, Materials Science and Engineering Department.

The project was conducted under the general supervision of Dr. W. F. Marcuson III, Director, Geotechnical Laboratory (GL), WES, and under the direct supervision of Dr. G. M. Hammitt II, Chief, Airfields and Pavements Division (APD), and Mr. T. W. Vollor, Chief, Materials Analysis Branch (MAB), APD. The WES Principal Investigator and preparer of this report was Dr. L. N. Lynch, MAB. Laboratory testing support was provided by Messrs. R. T. Graham and C. W. Dorman, MAB, and Mr. H. T. Carr and Ms. A. Williamson, Instrumentation Systems Division.

At the time of preparation of this report, Dr. Robert W. Whalin was the Director of WES. COL Bruce K. Howard, EN, was Commander.

CHAPTER - 1 INTRODUCTION

Sealant materials have been used throughout history for a multitude of applications. The applications range from the pitch material used in Noah's ark and the asphaltic materials used in ancient water canals to the fire-resistant sealants used in today's skyscrapers and the technologically advanced preformed seals used in the solid rocket boosters of the space shuttle. The required material properties of the sealant are as varied as the applications in which the sealants are used.

Joint sealant materials used in pavement applications are designed to protect the structural integrity of the pavement. The protection occurs through two functions; prevention of the retention of incompressible debris in the joint and the minimization of moisture infiltration through the joint into the base and subbase material. One reason joints are constructed in the pavement is to provide a stress relieving mechanism. As the temperature and moisture content of the pavement change, the pavement will expand or contract. The joints allow this movement to occur without damaging the pavement. The movement that occurs at the joint requires that the joint sealant material be flexible to accommodate the joint movement but rigid enough to resist the intrusion of debris.

Joint sealant materials began to be widely used in pavement applications during the early 1940's. Material specifications were developed during this time period in an effort to ensure the quality of the materials that were being used. The early specifications were based upon tests commonly used by the asphalt industry. Two potential reasons for the use of asphalt type tests were that the test procedures were familiar to those responsible for developing the specifications and the early joint sealant materials were produced from asphalt cements. As user agencies began to realize they needed joint sealant materials that were resistant to the solvent action of jet fuel and other chemicals, manufacturers began to use other base components such as coal tar to produce the sealant materials. Material specifications were developed to provide a quality control tool for these materials as well. Some of the problems associated with the specifications was the fact that they did not provide a direct correlation to field performance and direct comparisons could not be easily made between sealants that were manufactured out of different base materials. One

aspect of field performance that the material specifications attempted to address was aging. The aging of sealant materials was accelerated in the laboratory by exposing them to elevated temperatures for a specified length of time and then measuring the change in some physical property. However, no correlations were developed which indicate the amount of natural aging represented by the laboratory aging.

Although not specifically stated in the reviewed literature, the requirements used in the specifications were probably based on performance monitoring of field materials. It is assumed that the specification requirements were refined to eliminate the use of sealants that did not perform satisfactorily in the field.

It would be beneficial to the agencies that use joint sealant materials to develop an analytical tool that would allow them to directly compare different types of sealants on an equal basis. The developed methodology would need to include the viscoelastic nature and the aging characteristics of the sealant materials. Preferably, the methodology would also lend itself easily to numerical modeling methods such as finite element analysis.

Before a specific analytical methodology could be selected, it was important to understand what the desirable characteristics of a pavement joint sealant should be in order to provide satisfactory field performance. The ideal field molded joint sealant material would potentially have the following characteristics:

a) a low viscosity during insertion into the joint to allow adequate wetting of the concrete joint faces and adequate adhesion to the concrete joint face after the sealant has become a solid.

b) physical properties such as the stiffness of the sealant would be insensitive to the temperature extremes that it could be exposed to during field use or be positively affected by temperature changes (i.e., become more flexible when exposed to colder temperatures and stiffer when exposed to hotter temperatures).

c) resistant to oxidation, ultra-violet (UV) degradation, and other chemicals to which it may be exposed.

d) economical, from a life cycle cost basis and if possible an initial cost basis.

This research investigation attempted to address the viscoelastic nature of the sealants (item b), the changes in physical properties associated with aging (part of item c), and how the field performance of the sealant is affected by the viscoelastic nature and aging (item d).

Objective

The objective of this research was to develop an analytical methodology using dynamic shear rheology (DSR) that could be incorporated into current material specifications or used as the basis for a new material specification. The new material specification would assist user agencies in the selection of the most cost effective sealant for a given application. The methodology was developed using silicone sealant materials because the physical properties exhibited by silicone materials are typically less temperature sensitive than other types of sealants because of their chemical composition.

The methodology would include the use of finite element modeling to allow specific conditions of a given application to be modeled, thus providing a direct comparison between different sealants. In the past, researchers (as explained in Chapter 3) have used finite element modeling to evaluate sealant materials. Consequently, the development of a new finite element code for the sealant evaluation was not required. The previous investigations were parametric studies directed toward analyzing strains in the sealant material caused by varying joint configurations (both size and shape) or measuring the stresses that developed as the pavement joint expanded. The sealants were considered to be elastic and the properties were considered constant with age. These two assumptions are generally good immediately after the installation of the silicone sealant into the joint (i.e., when they are new); however, laboratory testing conducted at the U. S. Army

Engineer Waterways Experiment Station (WES) has indicated that silicone sealants will age and that materials produced by different manufactures will age differently. The viscoelastic nature or temperature sensitivity of the materials combined with aging characteristics were the two areas which required modification in the characterization methodology.

CHAPTER 2 - BACKGROUND

Design Function of Pavement Joint Sealants

Most of the distresses that could occur in a rigid pavement develop at the joints. The distresses could be caused by poor slab support, an erodible subbase or subgrade, excessive water, debris retention in the joints, poor load transfer, excessive traffic loads, sealant failure, slabs that are too long, dowel misalignment or corrosion, and/or soil movement under the slabs. The joint related distresses created by the above conditions include cracking, faulting, spalling, raveling, and excessive pavement movement (pumping or pavement growth due to incompressibles retained in the joint) [1]. Joint sealant materials and the practice of sealing joints to prevent the intrusion of water and/or incompressibles were developed to help protect the pavement structure from some of these distresses.

Potential Pavement Distresses

Water infiltration through a joint could cause the load bearing capacity of the pavement structure to be diminished. The diminished bearing capacity would occur because most subbase and subgrade materials become less stiff when they become damp or wet. Consequently, decreased stiffness could occur with changes in seasonal conditions. For example, in western Washington, falling weight deflectometer (FWD) testing has suggested that the modulus of well-graded base materials decreased by 25 percent from a high in the warm, dry summer months to a low in the cool, wet winter months. Subgrade materials at these same sites experienced a decrease of 15 percent from the summer to the winter [2]. Modulus values calculated in this manner are used in pavement design procedures, such as the one developed by the American Association of State Highway and Transportation Officials (AASHTO) [3], to develop a modulus of subgrade reaction (k-value) for the subbase and subgrade. Soil materials that exhibit greater losses of stiffness when they become wet, decrease the k-value and in turn increase the required thickness of the rigid pavement. Loss of support could be exhibited after construction by pumping, faulting, and eventually broken slabs.

Joint sealant materials are also designed to prevent the retention of in-compressible material in the joint. The stress relieving mechanism (i.e., the ability

of the pavement to expand and contract due to changes in ambient temperature or moisture content in the slab) of the pavement would be diminished and spalling could develop when incompressibles are retained in the joint. Incompressibles continue to fill the joint if joints are not properly maintained over the life of the pavement. This could result in pavement slabs pushing against one another and moving or sliding the pavement into unrestrained areas like the expansion joints before bridges. This slab movement is termed pavement growth. In most cases, expansion joints or terminal anchor lugs normally would contain the growth, however the growth has occasionally caused abutments to be split or tilted. In sever cases of pavement growth, a bridge could be pushed off the bridge seats [4]. Additionally, excessive pressures could buildup in slabs that are far away from a free end and those slabs could buckle or fail in compression to relieve the excessive pressure. The buckling of the slab would be very similar to the buckling action that occurs in a column under · compressive loading. Slabs nearer to the joint filled with incompressibles could be lifted off the subbase or subgrade causing the slabs to fail in flexure.

Forcing a bridge deck off the bridge seats and pavement buckling are associated with two of the more severe distresses. Spalling would be the more commonly occurring phenomena caused by the retention of incompressibles in the joint. Spalling could occur when excessive stresses build up at the joint faces due to the retention of incompressibles in the joint reservoir.

Types of Joint Sealants

There are numerous types of pavement joint sealant materials on the market today, and consequently there are several methods that can be employed to categorize the materials. The two major categories of pavement sealants are field-molded sealants and preformed seals. The most common preformed seal materials are compression seals that are generally extruded from a neoprene rubber material into various sizes and shapes by the manufacturer. Field-molded sealants, on the other hand, are liquid at the time of installation and conform to the shape of the

joint reservoir when they are installed. Each of these types of sealants or seals have advantages and disadvantages when being considered for use in the pavement joints. For this research, only field-molded sealants were considered.

Field-molded sealants can be further categorized by the method of application (hot-applied or cold-applied), the chemical component used as the base material of the sealant (asphalt cement, coal tar, silicone, nitrile rubber, polysulfide, polyurethane, etc.), a particular attribute of the sealant (jet-fuel-resistant (JFR)), blast resistant, low modulus, percent elongation, etc.), and/or the number of components used to obtain the final product (single, two, or multiple). Many material specifications used by the government and private industry categorize the materials by hot-applied (hot-poured) and cold-applied (cold-poured) followed by JFR or non-jet-fuel-resistant (non-JFR). In this report, sealants were categorized by the broad categories of cold-applied or hot-applied. More specific categories such as JFR were used when required.

Regardless of the type of sealant, the design function remains the same, to protect the pavement structure. When joints are properly sealed with a high-quality sealant as part of an aggressive pavement maintenance plan, all of the potential pavement distresses listed above may be mitigated. Additionally, the life cycle cost and the total maintenance costs of the pavement may be reduced.

<u>Field Failure Modes of Pavement Joint Sealants</u>

Joint sealant manufacturers estimated in 1991 that the total United States market for pavement joint sealant materials was approximately 100 to 125 million pounds (45 to 57 million kilograms) per year. If one assumed a joint reservoir size of 0.75 inch (19 mm) wide by 0.75 inch (19 mm) deep, the total linear meters of joints sealed each year would be approximately 356 to 445 million feet (108 to 136 million meters). With the cost of in-place joint sealant material ranging from $1.25 to $3.50 per linear foot ($4.17 to $11.67 per linear meter), the total expenditures for joint sealing projects in the United States would be between $445 million and $1.6

billion annually. Unfortunately, the field performance of field-molded materials continues to be inconsistent at best.

Field Investigations

The inconsistent performance of field-molded sealants has been highlighted by numerous "evaluation" studies. In 1967, a National Cooperative Highway Research Program (NCHRP) was conducted to investigate the practices and materials used to seal joints and cracks in pavements [4]. Part of this study included a survey of the state Departments of Transportation (DOT). Seventy percent of the survey respondents felt that the retention of incompressibles in the joints was a more serious problem than water infiltration. Therefore, sealant failures that allowed debris retention in the joint were of paramount concern. The failures included adhesion (loss of bond between the sealant and the joint face), cohesion (a splitting of the sealant material), and the embedment or intrusion of debris in the sealant. The recommendations from this study were very similar to the recommendations presented in many of the more current studies. The recommendations included;

a. Education of and communication between designers, laboratory testing personnel, manufacturers, and contractors.

b. More precise determination of the causes of existing sealant failures.

c. Development of meaningful test methods to evaluate new materials as they are introduced onto the market.

Some specific areas mentioned in the report to assist in developing meaningful tests to determine field performance included adhesion studies, work hardening investigations, fatigue analysis, and the determination of the optimum force-elongation-adhesion relationship. All of these areas of investigation for meaningful test methods concern better material characterization.

In 1982, NCHRP initiated a study to review the materials and techniques used to reseal joints and cracks [5]. The objectives of the investigation were to identify materials and techniques, determine costs, and determine benefits associated with resealing, and develop criteria for resealing if it was found beneficial. The

investigation concluded that resealing joints and cracks was generally beneficial, but the cost effectiveness of the maintenance activity had not been documented. It also stated that typical joint sealant failures included adhesion, cohesion, hardening (loss of elasticity) of the sealant caused by oxidation or weathering, and extrusion of the sealant from the joint. Therefore, from approximately 1967 to 1982, the basic types of sealant failures of field-molded pavement joint sealants reportedly remained the same. Furthermore, one recommendation of the NCHRP investigation was that more information was needed concerning the effective life of various sealant materials and the effects of differing placement techniques and climates on the effective life. One method for obtaining this type of information would be through material characterization techniques combined with finite element analysis.

A research project was conducted by the U.S. Army Engineer Waterways Experiment Station (WES) from 1985 to 1988 [6] to investigate military base personnel complaints concerning the field performance of pavement joint sealants. The military personnel complained that joint sealant materials used in resealing projects that were conducted according to specification, i.e., sealant material conformed to the appropriate specification, the joints were properly prepared, and the sealant was properly installed, were exhibiting poor field performance. The complaints were investigated using both laboratory evaluations of sealant materials and field surveys to determine the actual field performance of sealants and the procedures used by contractors to install the sealants. Most of the sealant failures noted in this study were adhesive, cohesive, and embrittlement caused by oxidation and weathering. The main conclusion of the study was that the failures in the field could not be directly related to material or project specifications. This conclusion was not drawn because the material and project specifications were found to be correct. Instead, the lack of correlation was because most of the projects in which poor field performance had occurred, either the sealant had not been tested by an independent laboratory for specification conformance, the project specifications had not been followed, or both. Comparisons between the laboratory evaluation and the

field survey did suggest that most of the failures in the field were similar to the more common types of failures noticed in material specification testing. Two of the tests most often failed during specification testing were the bond to concrete, which potentially can qualitatively indicate adhesive and cohesive failures, and the aged resilience, which can provide an indication of the effects of aging on the material. However, a direct correlation between these two tests and field performance could not be determined.

One final study that will be discussed before describing the potential mechanisms of the different failure modes is the Strategic Highway Research Program (SHRP) project H-106 "Innovative Materials Development and Testing, Volume IV - Joint Seal Repair [7]." This portion of the H-106 project was a full-scale investigation into the performance of the procedures and materials used in resealing joints in PCC pavements. The primary objective was to evaluate the relative performance of sealants based upon carefully designed and controlled field installations. Additional objectives were to determine the effects of sealant configuration and installation procedures on performance and to identify laboratory tests and material properties that correlated with field performance. In the Spring of 1991, approximately 1,600 joints were sealed. The joint test sites were located in five different states spanning four climatic regions. Evaluations of the field performance of the sealants were conducted at 1, 5, 9, 12, and 18 months. The observations based on these limited evaluations indicated that a total of 1.7 percent of the total length of the resealed joints experienced partial-depth adhesive failures. Most of these failures occurred at one test site in which residual joint sealant material was left in the joint before resealing. Other types of failures including partial-depth spalling of the joints occurred but in general the sealants were performing well. One last conclusion made in the investigation was that penetration, resilience, stress at 150 percent elongation, immersed elongation, and ultimate elongation "may be slightly correlated with adhesion loss in the field after 18 months [7]." Additionally, the researchers believed that the correlations could increase as time progressed.

Adhesive and Cohesive Sealant Failures

In each of the above mentioned investigations, the majority of sealant failures were associated with an adhesive or cohesive type failure. Therefore, these failure modes will be discussed in greater detail. Adhesive failures are usually attributed to improper joint preparation before inserting the sealant, an improper shape factor (depth to width ratio of the sealant material), use of an inferior material, and/or improper preparation of the sealant material before or during installation. Improper joint preparation basically means that some type of debris, either dust, curing compound, old sealant, etc., was not removed from the joint walls. A study conducted by the Naval Civil Engineering Laboratory (NCEL) in 1961 [8] concluded that in general for a joint sealant to obtain adequate adhesion to the joint faces, any oil or fuel deposits, curing compounds, and residual joint sealant must be removed. In addition, the study recommended that the joints be as dry as possible before sealing. The types of failures caused by improper joint preparation could be minimized by educating the contractor and inspection personnel involved with the project.

The definition of improper sealant preparation will depend on the type of material that is being used and the failure could be adhesive, cohesive or debris retention. When considering hot-applied materials, improper preparation could be over or under heating the sealant, heating the sealant for an extended period of time, or not completely cleaning the sealant melter at the end of each day. The hot-applied materials are typically an asphalt cement or a coal tar based material. These materials are subject to embrittlement through the loss of volatiles which could occur by over and/or extended heating. Under heating the sealant could prevent the viscosity of the sealant to decrease sufficiently to allow wetting of the joint face. Additionally, some of the hot-applied sealants are thermoset products that must be heated to the specified temperature to initiate cure. The under heating phenomenon would be more likely to allow debris embedment in the sealant than led to adhesive failures. Improper sealant preparation of cold-applied materials are usually limited

to the two-component sealant systems. In the two-component system, Component A (the accelerator) would be added to Component B (the base resin) in a specific proportion either by hand mixing or machine mixing. The most common problem that arises would be the incorrect proportion of the two components. If too much accelerator was used, the sealant would usually become brittle and the failure mode could be adhesive or cohesive. If not enough accelerator was used then the sealant could not reach adequate cure and the failure mode would typically be debris retention. Most of the above concerns would not present a problem if a single-component, cold-applied material was used because they are not mixed or heated. Instead, the potential installation problem associated with the single-component, cold-applied sealants would be installing them after the shelf life has expired. Education of the contractor and inspection personnel as well as closely monitoring the application equipment could minimize the problems associated with improper sealant preparation.

Two potential causes of adhesive failure that can be better understood by material characterization are also potential causes of cohesive failures. The two potential causes are an incorrect shape factor and the selection an inferior material. An inferior material in this instance may be one that is not capable of withstanding the amount of joint movement to which it will be exposed or it may be a material that excessively ages when exposed to environmental conditions.

Laboratory Investigations

The term shape factor was first used to describe the ratio of the depth of the sealant material in the joint to the width of the sealant in the joint in 1962 [9]. This concept can be visualized from figure 2.1. In this figure, the depth of the sealant is represented by D and the original width of the sealant is represented by W. The width of the sealant will be the same as the width of the joint at the time of sealant installation.

The idea concerning the shape factor of a joint sealant material began to receive attention in the early 1950's when consideration was given to the fact that a

relationship should exist between joint movement (changes in joint width), the original joint width, the material capabilities of the sealant material, and the performance of the sealant material [10]. Discussions concerning these considerations led to investigations into the development of a test that would model the joint. As work progressed on joint modeling tests, researchers began to realize that the depth of the sealant material in the joint and the temperature to which the sealant was exposed were also an important variables. Tons [11] made several recommendations and conclusions in this early research. Some of the conclusions and recommendations included:

a) The shape factor required for a specific project will depend upon the sealant material used, the minimum joint width, and the total joint movement.

b) Investigate the influence of temperature on the maximum allowable strains. Most of the tests conducted by Tons were ran at 80°F (26.7°C) or 0°F (-17.8°C). Tons stated that the allowable strain generally decreased for the materials tested at 0°F (-17.8°C) as compared to 80°F (26.7°C).

c) Investigate the adhesion properties of the sealant to the joint face.

d) Accumulate joint movement data from differing climatic regions.

e) Develop a durability test for sealant materials.

f) Define the types and shapes of sealants required for good performance to allow the manufacturers to meet those needs.

Many of the above recommendations infer that sealant materials are viscoelastic in nature, i.e., they exhibit stress relaxation and creep behavior which are dependent on time and temperature. The viscoelastic nature of sealant materials was further emphasized by Schutz [9] when he listed "Eleven factors to consider in designing a joint subjected to movement." Specifically, Schutz noted that the force required to stretch a sealant material will increase as the temperature decreases. Therefore, the lowest anticipated service temperature should be used as one selection

criteria when considering a sealant material. Schutz also noted that the hardness of the sealant material will vary with service temperature and that the anticipated joint movement (both amount of extension and rate of extension) must be considered when selecting a sealant.
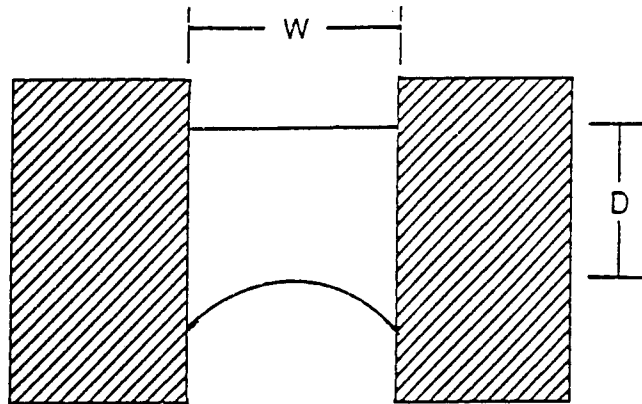
Figure 2.1: Typical Joint Sealant Configuration Demonstrating Shape Factor.

In 1965, Cook [12] investigated the viscoelastic properties of a polysulfide sealant by first·considering it as perfectly elastic and then considering its viscous nature. Because sealant materials can be formulated to have a wide range properties, Cook stated that both cases should be investigated. Cook suggested in this investigation that additional research into the effects of work hardening, weathering, and aging on the physical properties of a sealant was needed.

The investigations by Tons [11] and Cook [12] indicated that some of the failures that were being experienced in the field could be due to improper joint design. These designs did not consider the viscoelastic nature of the sealants and therefore the sealants were being installed into a configuration that was almost destined to cause failure. Based on the results of Tons' work [11], many current guide specifications require a shape factor of 1 to 1.5. However, sealants that gained widespread use after these studies, such as silicone sealants, require smaller shape

factors to perform satisfactorily. Tons realized that the shape factor would be dependent on the sealant material used. But many of the current user agencies and specification writers have apparently overlooked this fact as evidenced by a blanket requirement for a shape factor of 1 to 1.5 in many specifications. When an inappropriate shape factor is used, the strains in the sealant can exceed the critical value for the material leading to failure.

Material Deficiencies

The definition of an inferior material, when discussing joint sealant failures, has included a broad range of areas. Some specific areas or items include;

a) Materials that do not conform to the appropriate material specification.

b) Materials that exhibited failure because they could not accommodate the joint movement to which they were exposed (assuming the sealant was installed using the correct shape factor).

c) Materials that aged so dramatically that the sealant matrix deteriorated to a point that it could not adequately seal the joint.

The idea that an inferior material was one that did not conform to the appropriate specification will not be considered in this report for two basic reasons. Theoretically, sealants that do not meet specification requirements should not be used on a project; therefore, specification non-conformance should not be a cause of sealant failure in the field. A more realistic reason for eliminating specification non-conformance from the inferior material definition was that the majority of studies that have been conducted have not been able to prove a correlation between material specification compliance and field performance. Sealant failures that occur because an inappropriate shape factor was used is not the result of an inferior material. These types of failures are a direct result of poor design or construction practices. Therefore, the definition of an inferior material was considered to be one that ages or weathers to such an extent that it can no longer seal the joint or one that cannot accommodate the joint movement to which it was exposed.

The ability of a sealant material to function properly, both with respect to joint movement capabilities (the sealant working range) and aging degradation are directly related to the chemical constituents used to manufacture the product. Adding to the complexity of joint sealant working range and degradation kinetics are the facts that the materials are viscoelastic and chemical changes that could occur during aging or weathering could significantly affect the physical properties of the sealant.

Normally, when one considers the aging characteristics of a material, the perceived result would be a hardening of the material. This will generally be the case as sealant materials age. The aging process that could occur in sealant materials will be dependent upon the base constituent used to manufacturer the material and if the final product is a thermoset or a thermoplastic. Typical types of processes that could induce aging or weathering and degradation of polymeric materials are thermal, thermal oxidative, chemodegradation such as acid or solvent exposure, mechanical cycling, biochemical, or microbiochemical, photochemical, and ultra-violet (UV) exposure. In normal applications, the actual cause of aging and degradation will potentially be a combination of several of these processes occurring simultaneously.

In rubber materials, heat aging methods have been used to accelerate the aging process and thereby provide an evaluation of mechanical property changes that occur due to thermal oxidative processes. Normally the reference test used for the aged and unaged specimens would be a tensile test in which the moduli and ultimate properties are compared. The problem that arises with these types of tests is that the mechanical properties can be changed by varying the aging conditions [13]. Because the tensile properties will vary with conditioning, laboratory and field correlations for rubber materials like those used in automobile tires has remained ambiguous. The relationship between accelerated aging and the tensile properties of rubber materials has been mentioned here because silicone pavement joint sealant

materials are room temperature vulcanizing (RTV) materials, therefore, it was expected that various aging regimes would affect the material properties differently.

Silicone Sealant Materials

Silicone pavement joint sealants are generally single component materials whose major constituent is a polyfunctional siloxane. The typical curing mechanism for these materials is based on a blocked catalyst that is hydrolyzed by atmospheric moisture. This hydrolysis process liberates the active catalysts, thereby initiating cure [14].

The siloxane polymer used in the majority of the pavement joint sealants is hydroxy-ended polydimethylsiloxane. The chemical structure of the polydimethylsiloxane is shown below [15].

$$(CH_3)_2 \qquad (CH_3)_2 \qquad (CH_3)_2$$
$$| \qquad\qquad | \qquad\qquad |$$
$$HO \;-\; Si \;-\; O \;-\; (Si \;-\; O)_x \;-\; Si \;-\; OH$$
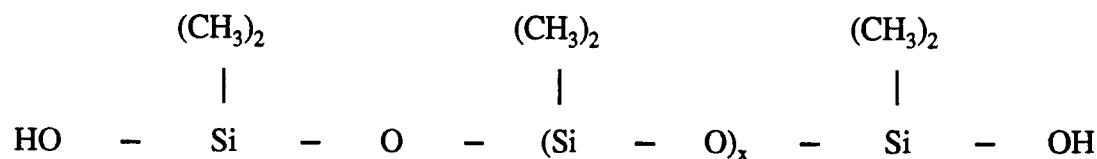
These polymers have a very low glass transition temperature (Tg) of approximately -189°F (-123°C) and they exhibit small viscosity changes with temperature. The lower temperatures can, however, retard the curing process. The Tg of an amorphous material is the temperature below which the material reversibly changes from a rubbery or viscous material to a brittle and hard material. Therefore, sealants that have low Tg values can potentially perform better than sealants with higher Tg values in climatic regions where the ambient temperatures are low.

Once the silicone material has cured, it can withstand temperatures of approximately 390°F (199°C) without decomposing. Therefore, the potential working range for the silicone sealants would be approximately -189°F (-123°C) to 390°F (199°C). The Si-O-Si backbone of the polymer is virtually unaffected by the effects of UV; therefore, silicone sealants are virtually weather resistant. It would appear from this brief discussion that silicone sealants would be the natural choice for most

sealing applications. However, some disadvantages do exist. The material cost for silicone sealants is higher than many of the other types of pavement sealants. Some argue that the installed costs of the various materials are similar, but one study has indicated that the installed cost of silicone sealants is approximately twice as much as asphalt based sealants [16]. Some of the silicone sealants have low tear resistance and some require a primer to achieve the desired adhesion. Tear resistance would usually be addressed in the sealing of pavement joints by requiring that the sealant be recessed in the joint. This would help protect the sealant from abrasion caused by vehicle tires. Silicone sealants also require that the substrate be meticulously cleaned. However, many sealant manufacturers of all types recommend that the joint faces be thoroughly cleaned to ensure sealant adhesion. Therefore, this disadvantage would not be limited to silicone sealants.

The performance of the silicone sealant will be a function of the final composition of the sealant. The final composition will include the basic polymer, any filler material, and cross-linkers. Fillers are usually added to a silicone sealant to provide reinforcement, thixotropy, and or bulk (i.e., an extender). Fumed silica is often added to silicone sealant formulations to achieve the desired tensile strength. The general molecular structure of the fumed silica or sand is SiO_2. The surface of the fumed silica is partially covered with SiOH which is the same functional group that is present on the polymer ends. Therefore, this filler can be incorporated into the polymeric network using the same chemistry that connects the polymer ends [15]. The fumed silica will also provide a thixotropic effect which helps prevent the sealant from flowing out of the joint before it cures. The non-reinforcing fillers that are often added to silicone sealants are calcium carbonate, carbon black, talc, or other inorganic materials. The inorganic, mineral-like materials are generally selected as fillers because they can be added to the silicone without diminishing the intrinsic desirable properties of the silicone sealant. Organic fillers are added to some silicone sealants to enhance specific properties or to reduce the material cost of the sealant, but these fillers often effect other material properties. Thermal stability

would usually be the property that would be diminished by the addition of organic fillers [15].

The cross-linking systems generally incorporated into many of the RTV one-component silicones involve a condensation reaction. The by-product evolved during the reaction would then be incorporate into the final sealant product and affect its properties. The most common types of cross-linkers are silane materials. The benefit of using a silane material is the fact that silanes are adhesion promoters and their silicone reactive ends are easily incorporated into the Si-O-Si backbone. Some of the silicone cross-linking systems involve an addition reaction. Klosowski [15] notes that in these systems, the Si-C-C-Si bonds are almost as stable as the Si-O-Si bond. Therefore, the desirable intrinsic properties of the silicone sealant remain intact.

Catalysts are used in silicone sealants to control the cure rate. Some common catalysts used in silicone sealants are metal carboxylates, alky metal carboxylates, and alkylmetal alkoxides. While manufacturers do not typically reveal the catalyst used in a specific sealant, some general trends concerning catalysts can be made. For example, increased catalyst concentration will increase the curing rate. There is a maximum limit to the amount of catalyst that can be added to the sealant. Above the maximum limit, sealant stability and specifically thermal stability can be diminish [15].

In the balancing act to formulate a reasonably priced silicone sealant that will perform satisfactorily, there are several items that can affect thermal stability. The reduction in thermal stability can decrease the amount of strain that the sealant can withstand as it ages. It will therefore be necessary to include thermal aging in the material characterization testing.

<u>Sealant Specifications</u>

Historical Review

The first significant quantities of rigid pavement used as a wearing course in the United States was around 1910 to 1915, but their use extends back into the late

1800's [17]. These early pavements had a plethora of problems. The problems included lack of standardized specifications, a general lack of understanding concerning the material properties of portland cement and portland cement concrete (PCC), and unenforced quality control procedures. However, one of the primary problems associated with these early pavements were that they were made with naturally occurring cements. Consequently, the early rigid pavement wearing courses had low compressive strengths, no transverse or longitudinal joints, poor construction techniques, and inadequate mix designs [10]. However, by 1912, work had been initiated to develop a joint that could withstand the traffic loadings to which the pavement was subjected [4]. The objective of the early pavement engineers was to simply "get the country out of the mud [4]." Joint sealant or filler materials used during this early construction period were primarily sand, tar paper, coal-tar pitch, asphaltic compounds, and wooden blocks. Material specifications for these products were very crude or non-existent. The earliest materials used in pavement joints were filler materials instead of sealant materials. The difference being that filler materials filled the joint to help prevent incompressible debris retention, but they did not necessarily adhere well to the PCC to prevent water infiltration.

By the 1940's, the trial and error methods used to construct rigid pavements had begun to give way to more standardized construction procedures and some of the emphasis of pavement performance was being focused on the joint sealant material. The emphasis on sealants came toward the end of World War II when communities were faced with rehabilitating existing factories and plants and providing for the rapid growth of traffic [4]. Hot-poured, rubberized asphalt compounds were the most predominate type of joint filler used during this period and it was also during this period that some of the first joint sealant material specifications were developed. Federal Specification (FS) SS-F-336, issued in May, 1942, was one of the first pavement sealant or filler material specifications. But this first specification was only a slight improvement from the no specification era. Problem with this specification were that it did not provide a consistent heating rate or heating time for sample

preparation. Since the sample preparation was not standardized, the results obtained when testing a material to this specification were not reproducible [18].

The need for more meaningful specifications became even more apparent after World War II when joint sealant manufacturers began an aggressive promotional program of their products. The field performance of the sealants did not live up to the promotional claims touted by the manufacturers, in fact the field performance of the rubberized-asphalt sealants was only marginally better than neat asphalt cement [18]. Some of the sealant manufacturers believed that the majority of the failures in the field were caused by improper heating of the materials during application. Two of the manufacturers addressed the perceived field heating problem by developing an indirect fired melter. The melter was designed using a double-boiler concept with an oil bath as the heat transfer media. A smaller version of the melter was designed to prepare laboratory samples for specification testing. This laboratory melter was first referenced in FS SS-S-164, issued in February, 1952, superseding FS SS-F-336. But full drawings and a description of the melter were not included in the sealant specifications until March, 1968, when the Interim FS SS-S-1401 was issued, approximately 25 years after the first specification. FS SS-S-1401C is the current Government specification that is used to specify hot-applied, non-fuel-resistant, sealants for military projects. This specification has remained virtually unchanged for the past 25 years.

As joint sealants were used on more diverse types of pavements like airfield parking aprons, etc., another problem became evident. The problem was that the rubberized-asphalt sealants dissolved when fuel was spilled on them. Industry and government agencies began an effort to meet this need in the late 1950's to early 1960's by developing a material specification for jet-fuel-resistant sealants. Federal Specification SS-S-167, issued June 1956, was the result of this effort. Federal Specification SS-S-1614, issued November 1969, superseded FS SS-S-167 and FS SS-S-1614A is the specification currently used to specify hot-applied, jet-fuel-resistant sealants for military projects. As with FS SS-S-1401C, the requirements in FS SS-S-

1614A have also remained virtually unchanged since the original version. This brief history of the development of joint sealing specifications indicates that any technological advancements that may have been made in the sealant industry have not been addressed by the specification writers. Consequently, user agencies have been slow to accept technological advancements made in the joint sealant industry.

The American Society for Testing and Materials (ASTM) also began to develop pavement joint sealant material specifications in the late 1950's to early 1960's. The developmental history of these specifications will not be discussed because the test procedures required in the ASTM specifications are very similar to those used in the Federal Specifications. Table 2.1 summarizes the test methods used in the current versions (as of July 1995) of Federal and ASTM specifications. Table 2.2 provides general characteristics and generic uses for sealant materials.

Two items become apparent as one reviews the various specification test procedures and the general characteristics provided in Tables 2.1 and 2.2. First, the test procedures that are listed provide information concerning the physical properties of the sealants but direct correlations between these test procedures and the actual performance of the sealant in the field is not evident. This means that simply because a sealant material conforms to the appropriate material specification, it will not necessarily perform satisfactorily in the field. In fact, most field evaluations conducted by the WES have indicated that pavement joint sealant material failures could be attributed to poor construction, inferior materials, and/or a lack of inspection by the user agency [6, 19]. Poor construction, in this case, could refer to improper project specification requirements or actual construction procedures used by the contractor. Inferior materials, as described in the referenced report, implied that no testing was conducted on the sealant material or that the material was improperly installed into the joint. When specification was not conducted on the lot number of sealant used on the project, specification conformance could not be verified. However, there were some projects where all procedures were adhered to and the sealant material failed within a very short time after insertion into the joint.

Table 2.1. ASTM Specification Test Procedures For Field-Molded Pavement Joint Sealants.

Test Procedure[1]	ASTM D1190	ASTM D3405	ASTM D3406	ASTM D3569	ASTM D3581	ASTM D5078	FS SS-S-1401	FS SS-S-1614	FS SS-S-200
Nonimmersed Penetration	✓	✓	✓	✓	✓	✓	✓	✓	N/A
Fuel-Immersed Penetration	N/A	N/A	N/A	✓	N/A	N/A	N/A	✓	N/A
Flow	✓	✓	✓	✓	✓	✓	✓	✓	✓
Nonimmersed Bond	✓	✓	✓	✓	✓	N/A	✓	✓	✓
Water-Immersed Bond	N/A	N/A	✓	✓	✓	N/A	✓	✓	✓
Fuel-Immersed Bond	N/A	N/A	N/A	✓	✓	N/A	N/A	✓	✓
Unaged Resilience	N/A	✓	✓	✓	N/A	✓	✓	N/A	✓
Aged Resilience	N/A	N/A	✓	✓	N/A	N/A	N/A	N/A	✓
Artificial Weathering	N/A	N/A	✓	✓	N/A	N/A	N/A	N/A	✓
Tensile Adhesion	N/A	N/A	✓	✓	N/A	N/A	N/A	N/A	N/A
Solubility (Change in Weight)	N/A	N/A	N/A	✓	✓	N/A	N/A	✓	✓

[1] Consult the appropriate specification for the specific test procedures and test temperatures, they are not identical between the various specifications. Not all test requirements for all specifications were listed.

Table 2.2. General Characteristics and Generic Uses for Sealant Materials.

Specification	General Characteristics[1]	Generic Use
ASTM D 1190	Asphalt based, hot-applied elastic-type sealant containing ground rubber and/or polymeric materials and reinforcing fillers. Supplied in solid form from the manufacturer.	Sealing joints and cracks in ACC[2] and PCC[3] pavements. Not intended for use in areas where fuel spillage is expected.
ASTM D 3405	Asphalt based, hot-applied sealant containing ground rubber, and/or plasticizers, and reinforcing fillers. Supplied in solid form.	Sealing joints and cracks in ACC and PCC pavements. Not intended for use in areas where fuel spillage is expected.
ASTM D 3406	Coal tar based, hot-applied sealant containing plasticizers and fillers. Supplied in either solid or liquid form. The liquid form undergoes cross-linking during the heating process to become a solid.	Sealing joints and cracks in PCC pavements. The sealant should not be used in ACC pavements due to potential incompatibility problems between coal tar and asphalt cement products.

[1] The base component provided in this column is generally what is used by the industry to manufacturer sealants conforming to the listed specification. It does not preclude other base component materials being used to manufacture the sealant type.
[2] ACC = asphalt cement concrete
[3] PCC = portland cement concrete
[4] JFR = jet-fuel-resistance
[5] PVC = polyvinyl chloride

Table 2.2. (continued).

Specification	General Characteristics[1]	Generic Use
ASTM D 3569	Coal tar based, hot-applied, JFR[4] sealant containing PVC[5], plasticizers and fillers. Supplied in liquid or solid form. The liquid form undergoes cross-linking during the heating process to become a solid.	Sealing joints and cracks in PCC pavements where fuel spillage is expected. The sealant should not be used in areas where concentrated aircraft exhaust is expected. It should also not be used to seal cracks in ACC pavements due to potential incompatibility problems between coal tar and asphalt cement products.
ASTM D 5079	Asphalt based, hot-applied filler containing ground rubber and fillers. Supplied in solid form.	Filling cracks in ACC pavements. This material is not a sealant and should not be expected to perform in the same manner as a sealant. The material is not intended for use in areas where fuel spillage is expected.

[1] The base component provided in this column is generally what is used by the industry to manufacturer sealants conforming to the listed specification. It does not preclude other base component materials being used to manufacture the sealant type.
[2] ACC = asphalt cement concrete
[3] PCC = portland cement concrete
[4] JFR = jet-fuel-resistance
[5] PVC = polyvinyl chloride

Table 2.2. (continued).

Specification	General Characteristics[1]	Generic Use
FS SS-S-1401	Asphalt based, hot-applied sealant containing ground rubber, plasticizers, and/or polymeric materials, and reinforcing fillers. This material is similar to ASTM D 3405 materials and is supplied in solid form.	Sealing joints and cracks in ACC and PCC pavements. The material is not intended for use in areas where fuel spillage is expected.
FS SS-S-1614	Coal tar based, hot-applied sealant containing PVC, plasticizers, and fillers. Similar to ASTM D 3569 materials. This material can be supplied in solid or liquid form.	Sealing joints and cracks in PCC pavements where fuel spillage is expected. The material should not be used to seal cracks in ACC pavements due to potential incompatibility problems between coal tar and asphalt cement products. The sealant is not intended for areas that will received concentrated aircraft exhaust.
FS SS-S-200	Cold-applied, two-component, JFR and heat resistant sealant. The material can be supplied in either a hand mix or machine mix formulation.	Sealing joints and cracks in PCC pavements that are subjected to fuel spillage and concentrated aircraft exhaust.

[1] The base component provided in this column is generally what is used by the industry to manufacturer sealants conforming to the listed specification. It does not preclude other base component materials being used to manufacture the sealant type.
[2] ACC = asphalt cement concrete
[3] PCC = portland cement concrete
[4] JFR = jet-fuel-resistance
[5] PVC = polyvinyl chloride

These "correct" projects are ones that delineate the need for test procedures that can be correlated to field performance.

An example of a test procedure that would appear to be related to field performance is the bond to concrete test. There are three bond to concrete tests; nonimmersed, water-immersed, and fuel-immersed tests. The water- and fuel-immersed tests are similar in that the specimens are submerged in a test fluid for a specified amount of time, conditioned at the test temperature (usually 0°F (-17.8°F) or -20°F (-28.9°C)), and tested. The nonimmersed bond test eliminates the immersion in test fluid before testing. The tests can identify materials that could potentially fail in the field but the test requirements are based more on an undocumented historical database, i.e., the developers of the specification believed that materials which possess the properties to pass the test would perform satisfactorily in the field. Additionally, the bond to concrete test is generally conducted by pouring the sealant material between two concrete blocks that are spaced 0.5 inch (12.5 mm) apart. The sealant reservoir formed by the concrete blocks is 0.5 inch (12.5) wide by 2 inches (50.8 mm) deep by 2 inches (50.8 mm) in length. This configuration creates a shape factor of four. Studies by Tons [11] indicate that this shape factor is extreme and that shape factors of 1 to 1.5 would be more appropriate for satisfactory field performance of the types of sealants listed in Tables 2.1 and 2.2. It is not uncommon for the test requirements in material specifications to be more extreme than the conditions the material will be exposed to in the field. But to be a meaningful test requirement, it would be beneficial if that test could be correlated with field performance of the material. Some problems potentially exist with the other test methods and requirements listed in Table 2.1, but the technical arguments are similar in nature, and, therefore, will not be discussed.

Silicone Sealant Specifications

The second item that becomes apparent when examining Tables 2.1 and 2.2 is the fact that silicone based sealants are not included in any of the ASTM or Federal specification concerning pavement joint sealants. Instead, most of the

material specifications that have been developed for materials like silicone sealants are specifications for building sealants.

The lack of an industry accepted materials specification for silicone pavement joint sealants lead many user agencies to develop internal material specifications. The U.S. Army Corps of Engineers, the Federal Aviation Administration, the U.S. Departments of the Army, Air Force, and Navy, the majority of the 50 state DOTs, and every silicone sealant manufacturer has developed or adopted some type of specification. In addition, the time required to complete all of the testing required in any one of the specifications is approximately 6 to 8 weeks. Table 2.3 provides some of the typical tests used in silicone specifications and some selected silicone specifications have been included in Appendix A. With the proliferation of specifications, and the amount of time required to determine if a material actually conforms to the selected specification, many user agencies will specify another type of sealant material and then allow the use of a silicone material as an "experimental" application or they will accept a manufacturer's certificate of certification.

The lack of an industry accepted materials specification has not prevented the use of silicones in pavement applications, but it has possibly resulted in a slower market growth of the materials. Some of the first reported uses of silicone pavement joint sealants were in 1954 [20], when the New York State Department of Public Works began to investigate materials that could be used to seal the expansion and contraction joints in bridges. Two silicone sealants were used in this investigation. One of the silicones employed the use of a "liquid accelerator or catalyst" and it reportedly failed in adhesion and cohesion "in less than a month" [20]. Additional information was not provided for the other silicone material but it reportedly produced an effective seal for two years. At the end of two years, the material was removed from the joints. The reason for removing the sealant was not given. The biggest complaint reported concerning the cold-applied sealants used in the study was that they required the use of a primer and they were difficult to install. Most other investigations conducted during the late 1950's through the 1960's simply noted that there were cold-applied materials used but these materials were not discussed in

Table 2.3. Specification Test Procedures Recommended by Silicone Sealant Manufactures.

Test[1]	Test Method
Appearance	Visual Observation
Extrusion Rate (grams/minute)	MIL-S-8802[2]
Specific Gravity	ASTM D 1475
Flow (maximum inches)	ASTM D 2202
Tack Free Time (minutes)	ASTM C 679
Durometer[3] (Shore A)	ASTM D 2240
Modulus at 150% Elongation (maximum psi)	ASTM D 412, Die C
Elongation[3] (minimum percent)	ASTM D 412, Die C
Adhesion to Concrete (minimum percent elongation)	ASTM D 3583
Movement (tension and compression)	ASTM C 719
Accelerated Weathering	ASTM C 793

1 The specific title of the test may vary between the different manufacturers and the test procedures may be modified from the procedures listed in the referenced specification.

[2] MIL-S refers to Military Standard specification.

[3] Samples usually cured for 7 days at $77 \pm 2°F$ ($25 \pm 3.2°C$) and $50 \pm 5\%$ relative humidity.

detail. A 1967 study conducted under the auspices of NCHRP [4] indicated that many "exotic" materials such as polysulfide, urethane, and other elastomeric materials were developed in the 1950's. However, the performance of these materials was less than satisfactory because, as the investigation reports, the materials were placed on the market prematurely and contractors made mistakes installing the sealants. The mistakes were made because they were not familiar with these new materials. The poor performance of these elastomeric materials lead many states including Kansas, Nebraska, New York, Pennsylvania, and Rhode Island, to either totally reject the use

of elastomeric sealants or allow their use only in restricted applications. The report noted only two agencies that allowed the use of elastomeric sealants, Hawaii and the Province of British Columbia. Silicone sealants were not specifically mentioned in the NCHRP report. Their lack of inclusion by name implies that either they were not available for installation or their performance was also unsatisfactory.

Silicone sealants began to penetrate the building sealant market in the mid-1960's [21] and the pavement sealant market in the 1970's. The latter conjecture is based upon Case History documentation from the Dow Corning Corporation. These Case Histories indicated that silicone sealants began to be installed in highway joints between 1973 and 1977 [22, 23]. In a field evaluation of a low modulus silicone highway joint sealants conducted in 1984 [24], the earliest installation date listed was in Georgia in 1977. This coincides with the data presented in the Case Histories. The field evaluations of the low modulus silicone material indicated that these materials were apparently performing well in various climatic regions around the country. Many user agencies began to conduct field tests using silicone sealants and most were very satisfied with their field performance.

The promising reports from the first field evaluations lead some user agencies to believe that their sealing problems were finally over. However, the user agencies wanted a generic silicone specification that their designers could insert into joint sealing and resealing contracts. During the early 1970's, ASTM formed a task group to develop such a specification. As of August 1995, this specification had not been completed. Frustration concerning the specification development lead to the proliferation of independent specifications and the lack of standardization that currently exists.

Most of the silicone specifications that have been developed were modeled after building sealant type specifications. The use of building sealant specifications as a guide is understandable because silicone sealants were first used in the building industry; therefore, these were the tests the manufacturers believed properly characterized the silicones. Typical specifications that were developed for hot-applied sealants were based upon asphalt cement type tests like penetration. These

tests were not satisfactory for the silicone materials because the silicones had to be exposed to the atmosphere to cure. Silicone sealants inserted into a container for penetration type testing would practically never cure. This was verified by a study conducted at WES [19]. Silicone sealants were poured into six-ounce (0.18 liter) containers as required for penetration and resilience testing. After three months of laboratory exposure, only the top 0.25 to 0.5 inches (6.4 to 12.5 mm) had cured. Inability to properly cure the test specimens implied that many of the standard pavement sealant test procedures could not be used in a silicone specification and the excessive length of time required to test a sealant to the building sealant specifications was unacceptable to some user agencies and contractors.

Correlations between field performance and the laboratory results obtained from material specification testing is also a problem with silicone sealants. Studies have not been conducted that would allow someone to predict the field performance of the material based on a test or series of tests that are listed in the specifications. Because of the lack of standardization between the various silicone specifications and user agency desires to determine if a sealant material will perform satisfactorily within a given set of conditions, a combination of material characterization testing and finite element modeling was needed. The material characterization test or tests should be relatively quick to provide user agencies and manufacturers with the ability to know within days if a material is satisfactory instead of months. Some arguments have been presented which indicate that the time consuming tests are more realistic of field conditions and should not be excluded simply for the sake of expedience. This may be true, but if the testing is so time consuming that they are never used, they do not provide any benefit. Additionally, some manufacturers point out that lengthy specification testing means that the sealant they produce will have to be inventoried until it has passed the required testing. The increased inventory would be expensive especially when they discover that after storing the material for two months that the material did not comply with the appropriate specification.

Qualified Product Lists and Product Acceptance

To help address some of these concerns, some manufacturers proposed that sealant materials be qualified on a yearly basis with samples being selected at random. These test results would then be used to develop a qualified products list (QPL). Contractors would then select a sealant from the QPL for a given project. There are many arguments that have been presented against the QPL plan. One argument present against the QPL plan was that some agencies cannot not use QPLs or they did not want to expend the effort and funds to establish such a program. Another argument introduced concerns over the consistency of the sealant materials that are manufactured. Laboratory specification testing had indicated that the physical properties between two lot numbers of the same brand of joint sealant material could be very different. Round robin tests conducted from 1992 to 1993 by ASTM on pavement joint sealant test procedures indicated that the deficiencies could lie within the actual tests as well as the materials. The data from the round robin indicated that the results of sealant specification testing can be contradictory from one laboratory to another. Similar findings were made in research conducted by Crafco, Inc., and WES [25]. The specific results of this study indicated that it was possible for two laboratories to test samples taken from the same lot or batch number and the material conform to specification requirements in one laboratory while failing in the other. It should be noted that failure of any one of the test requirements in a specification constitutes non-conformance.

Accelerated Curing

Many of the concerns listed above can be addressed by implementing material characterization tests such as rheological and thermal analysis. Test reproducibility will still be a concern that must be investigated. The implementation of these material characterization tests will not completely address the concerns regarding the time required to complete the tests. Manufacturers of silicone sealant material often recommend that the test specimens be allowed to cure for 14 to 21 days before certain tests are conducted. However, to better serve the user agency needs, it would be beneficial to reduce the 14 to 21 day cure time.

Most single-component RTV silicones do not reach full cure in the field until 14 to 21 days after installation. Therefore, there is some concern about what problems joint movement will cause in the uncured sealant. One potential problem is that the movement can cause thin spots to form in the sealant that can lead to premature failure. For example, if the silicone sealant is installed in the joint and then the joint opens, the center of the sealant bead thins. As the sealant begins to cure, this thinner portion will cure faster. As the joint then begins to close, the thin center of the sealant can be forced above the pavement surface and abraded by traffic. This type of failure is more of a problem when silicone sealants are used in the joints of buildings because these joints are usually subjected to much higher movements than pavement joints.

The current situation involving specification testing for military projects includes the contractor selecting the sealant to be used for the project and submitting it for specification testing. The samples are then sent to a government approved laboratory for testing. Actual testing takes 35 to 41 days. If the material does not conform to specification requirements, it is rejected and can not be used on the project. The contractor must then submit another sealant. The process often creates delays and can increase the overall cost of the project. By reducing the required testing time, these delays could be mitigated. Specific accelerated curing and aging procedures are discussed in Chapter 3.

Viscoelastic Nature of Sealants

Strains in Sealants

In 1959, Tons [11] proposed a mathematical model based on maximum strain calculations that detail the suspected relationship between joint movement and sealant configuration. The simplifying assumptions made by Tons were;

    a) The cross-sectional area of the in-place sealant was rectangular.

    b) The sealant was a liquid-type, homogeneous material that changed shape when extended and compressed but did not change volume.

    c) The top and bottom free surfaces deformed parabolically and equally as the sealant was extended and compressed.

d) There was no three sided adhesion, i.e., assumed that the sealant material did not adhere to the bottom of the joint reservoir.

e) The strain in the sealant along the parabolic surfaces was uniformly distributed during extension.

f) The minimum and maximum joint widths were the maximum strains the sealant would experience regardless of the width of the joint when it was sealed.

These assumptions were reasonable for a first approximation. However, sealants installed in the field would normally have a backer rod material against the bottom surface. Backer rod materials are used to regulate the depth of the sealant in the joint and to provide support to the sealant as it solidifies. Most of the backer rod materials are round, and, therefore, the inserted sealant would not be rectangular. Figure 2.2 illustrates the joint configuration assumed by Tons for determining strains in the sealant.

Tons noted that as the joint expands to some width from the minimum width, the cross-sectional area of the joint would be given by:

$$J_S = (W_X - W_{min}) * D_X \tag{1}$$

where

$$
\begin{aligned}
J_S &= \text{the cross-sectional area} \\
W_X &= \text{the width at any extension} \\
W_{min} &= \text{minimum joint width} \\
D_X &= \text{depth of sealant material in the joint reservoir}
\end{aligned}
$$

Since a liquid-type material was assumed, the cross-sectional area of the sealant would not change. Consequently, the area of the parabolic surfaces would be equal to the increase in the cross-sectional area of the joint reservoir and the maximum curved in depth, H, could be calculated as follows:

$$A_P = 0.5 * (W_X - W_{min}) * D_X \tag{2}$$

where

A$_p$ = area of one of the parabolas (area ABC)

H = the distance from the original sealant surface to the peak of the parabola.

To calculate H, the equation for the area of a parabola $(A_p = 2/3*H*W_x)$ would be substituted for A$_p$ and the equation would then be solved for H. The equation for calculating H would become:

$$H = 1.5 * \left(\frac{A_P}{W_X}\right) = \frac{0.75 * D_X * (W_X - W_{min})}{W_X} \qquad (3)$$

The strain that the sealant would undergo as the concrete contracted would not be directly proportional to the joint extension because of the parabolic deformation at the top and bottom surface.
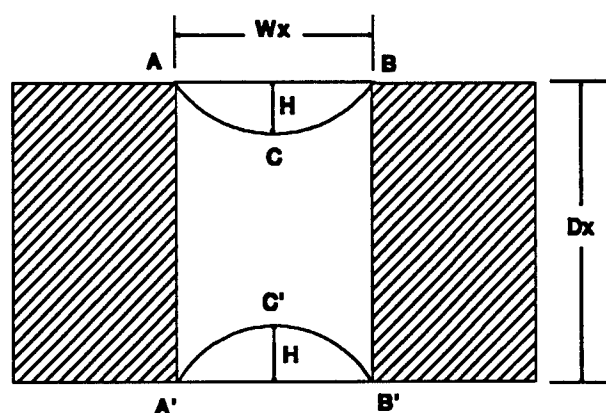
ACB = length of parabola

ACBA = area of parabola

Dx = depth of joint sealant

Wx = extended joint width

H = maximum recessed depth of sealant surface

Figure 2.2: Sealant Configuration.

The lack of proportionality could be illustrated by dividing the sealant into horizontal elements. As the cross-sectional area of the joint reservoir increased due to concrete contraction, the elements closer to the two free surfaces would have a greater increase. The elongation of the two free surfaces could be calculated by using the arc length for a parabola. Substituting in the variables from Figure 2.2, the arc ABC could be calculated by:

$$L=0.5*(W_X^2+16*H^2)^{0.5}+\frac{W_X^2}{8*H}*\ln(\frac{4*H+(W_X^2+16*H^2)^{0.5}}{W_X}) \qquad (4)$$

Using the parabolic arc length, the maximum strain of the sealant at the surface would then be calculated by:

$$S_{max}=(\frac{L-W_{min}}{W_{min}})*100 \qquad (5)$$

where

S_{max} = the maximum strain in the surface of the sealant

L = parabolic arc length (ACB from Figure 2.2).

Using these equations, a joint reservoir that had a minimum width of 0.5 inches (12.5 mm) and a sealant depth of 0.5 inches (12.5 mm) that was extended to 0.75 inches (19.1 mm) would have a maximum strain at the surface of 60 percent. The strain in the center of the sealant material would be approximately 50 percent. If the minimum joint width was reduced to 0.25 inches (6.3 mm), the strain in the center of the sealant would increase to 200 percent and the strain along the surface of the sealant would be approximately 274 percent. The calculations indicate that the maximum strain in the sealant material could be reduced by decreasing the depth of the sealant and/or increasing the minimum joint width. Therefore, the calculated maximum strain in the sealant material would depend on the minimum joint width, the amount of joint expansion, and the depth of the sealant material. This suggests that to minimize the stresses in a sealant for a given strain, a lower modulus material

would be desirable. This brief analysis assumed that all of the sealant materials have equal wetting characteristics with the concrete joint face and it highlights the fact that the maximum strain a sealant will be able to withstand must be determined.

Tons' [11] research into the relationship of joint movement versus the shape factor of the joint sealant provided a good foundation for future research. It also highlighted the need for a better understanding of the viscoelastic nature of sealants.

Stress Relaxation and Creep Behavior

Cook [12] investigated the interrelated phenomena of stress relaxation and creep by first investigating the sealant as a perfect elastic and then considering the viscous nature of the sealant. For the perfectly elastic material, Cook used a kinetic theory model developed by Treloar which described the internal work of deformation within a joint sealant as:

$$W = 0.5 * G(\alpha_1^2 + \alpha_2^2 + \alpha_3^2 - 3) \tag{6}$$

where

W = internal work of deformation within the joint sealant

G = shear modulus

α = ratio of extended length to the original dimension in each direction.

The external work done by the applied force would be:

$$dW = f_1 d\alpha_1 + f_2 d\alpha_2 + f_3 \alpha_3 \tag{7}$$

By differentiating the internal work equation and equating it to the external work equation, Cook developed two general stress-strain relations that are valid for the Gaussian probability function on which Treloar's statistical derivation was based. This means that the formulas would be valid for elongations up to approximately 200 percent. The two formulas are:

$$\alpha_1 f_1 - \alpha_3 f_3 = G*(\alpha_1^2 - \alpha_3^2) \qquad (8)$$

$$\alpha_2 f_2 - \alpha_3 f_3 = G*(\alpha_2^2 - \alpha_3^2). \qquad (9)$$

These equations can be further simplified when considering actual joint sealant problems because the joint sealant materials normally extend across two or more slabs. This implies that the sealant length in this direction would be at least 25 to 30 feet (7.6 to 9.1 meters) versus a cross-section area of approximately 1 to 2 square inches (25.4 to 50.8 square mm). The extension ratio in this direction (α_3) could therefore be considered unity. The sealant volume was assumed constant as it was extended; therefore, $\alpha_1 \alpha_2 \alpha_3$ must also equal unity which implies $\alpha_1 = \alpha$, $\alpha_2 = 1/\alpha$, and $\alpha_3 = 1$. Assuming that the only applied force acting on the sealant was in the tensile direction (α_1), the stress would reduce to [12]:

$$f = \frac{dw}{d\alpha} = G*(\alpha - \frac{1}{\alpha^3}) \qquad (10)$$

Therefore, if the sealant was perfectly elastic, the internal stress would be a function of the extension ratio in the tensile direction and the shear modulus of the sealant.

The next step in Cook's investigation was to consider the sealant material as a "flowing solid [12]." The term flowing solid illustrates the meaning of viscoelasticity from a more mechanical view point, i.e., a solid with some characteristics of a liquid. Conversely, a rheologist would typically think of viscoelasticity as the study of liquid materials that have some characteristics of a solid. From either view point, viscoelasticity implies that the material has a viscous component and an elastic component and the predominate component exhibited by the material (either viscous or elastic) will be dependent on the rate of the experiment or observation and the temperature during the observation. Macroscopically, viscoelasticity can be separated into three basic components; two of which are reversible and one which is irreversible. The three components described by Cook were [12]:

a)   Instantaneous elasticity which was a straightening out or uncoiling of the main polymer chain.

b)   Delayed elasticity which consisted of orienting the polymer chains in the direction of stress.

c)   Viscous flow which occurred as the polymeric chains begin to slip past one another due to the stress.

Therefore, to model a viscoelastic material, a minimum of two elements would be required; a Hookean spring ($\sigma = E\varepsilon$; where σ = stress, E = spring stiffness or Young's modulus, and ε = strain or elongation) to model the elastic portion of the material and a Newtonian dashpot ($\sigma = h(d\varepsilon/dt)$ where σ = stress, h = viscosity, $d\varepsilon/dt$ = strain rate) to model the viscous flow of the material. Cook used the Maxwell and Voigt models and combinations of these models to characterize the sealants. The Maxwell model combines the two idealized elements (spring and dashpot) in series as shown in Figure 2.3. The Voigt or Kelvin model combines the two elements in parallel as shown in Figure 2.4. The Maxwell model can be used rather effectively to model stress relaxation, but it will not model creep very realistically. The deficiency in modeling creep has been previously described [12, 26, 27]. The problem that occurs when solving for the creep condition is that $d\sigma/dt$ becomes 0 which implies that the stress, σ, at some time t is equal to σ_0. Therefore, the strain would continue to increase linearly with time instead of increasing at the beginning and reaching a plateau as has been observed in many creep experiments.

The Voigt model on the other hand can be used to characterize the creep response of a material but it does not model stress relaxation realistically [28]. The problem that occurs when solving for the stress relaxation condition is that ε is constant. Because ε is constant, the model predicts a linear relationship between stress and strain for all limits. This relationship is generally not observed in polymeric materials. It would appear, therefore; that to model both stress relaxation and creep, a combination of Maxwell and Voigt models would be required.

The simplest combination of the Voigt and Maxwell model would contain four elements. For most polymers, this combined model would be to simplistic because polymers exhibit multiple stress relaxation times. The multiple stress relaxation times will be dependent upon the multitude of ways in which the molecules of the polymer could regain their most probable configuration. The spectrum of these relaxations would need to be represented or approximated by a statistical distribution function. Cook noted that the type of sealant material used in the investigation (a polysulfide based material) did exhibit a single stress relaxation time as shown by earlier researchers [12]. Therefore, Cook could use the simplified model as an adequate approximation. The simplified four element model used by Cook is illustrated in Figure 2.5. The basic equation for the stress as put forth by Cook was:

$$\delta = (\beta\eta_1 + e^{-\frac{t}{\tau 1}} * (G_1\varepsilon_0 - \beta\eta_1)) + (\beta\eta_2 + e^{-\frac{t}{\tau 2}} * (G_2\varepsilon_0 - \beta\eta_2)) \tag{11}$$

where

β = strain rate

h_1, h_2 = viscous constants taken from the creep curve

G_1, G_2 = elastic constants taken from the creep curve

ε_0 = initial displacement

$\tau 1, \tau 2$ = relaxation times, defined as the time required for the stress in the sealant to decay to $1/e$ times the initial stress

t = length of time from start of test

The creep curves mentioned in the above variable definitions were obtained from experiments conducted by Cook [12]. In addition to creep tests, Cook also conducted stress relaxation, modulus of elasticity, and peel adhesion tests. Using the results from these tests and various potential scenarios to which a sealant could be exposed, Cook calculated the force acting at the sealant/concrete interface and the stress acting in the necked down portion of the sealant using the elastic material equations.

41

Figure 2.5: Simplified Four Element Model Used by Cook.

σ, ε

$\sigma_2 = \eta(d\varepsilon_2/dt)$

$\sigma_1 = E\varepsilon_1$

σ, ε
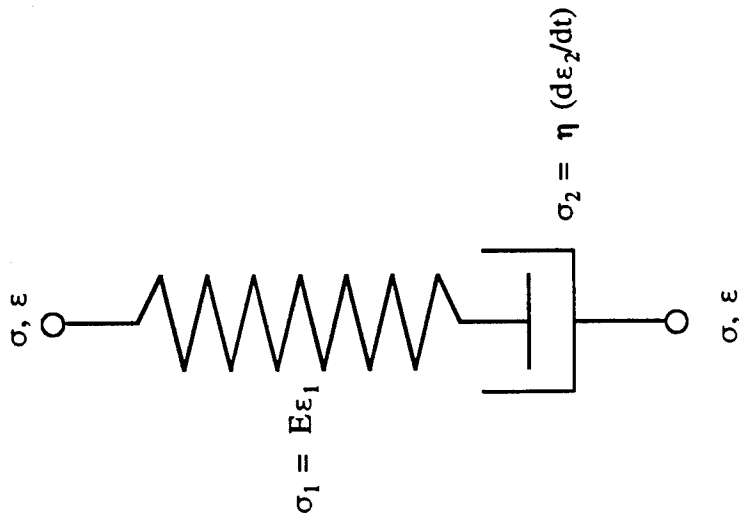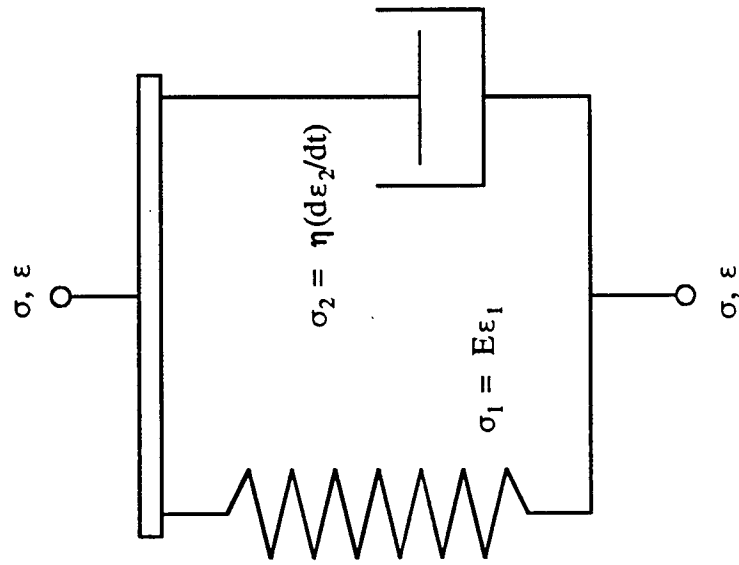
Figure 2.4: Voigt Model

σ, ε

$\sigma_2 = \eta\,(d\varepsilon_2/dt)$

$\sigma_1 = E\varepsilon_1$

σ, ε

Figure 2.3: Maxwell Model

Comparing these calculated results to the peel adhesion results and the stress-straincurves, Cook indicated the type of failure that the sealant should exhibit under the specified strain conditions.

The stress relaxation analysis conducted by Cook indicated that the major problems associated with sealant performance were the shape of the sealant and the ability of the sealant to recover its shape instead of stress. This conclusion was based upon the fact that the stresses in the sealant material dropped to almost negligible values within a very short period of time. Cook reported a 20 percent reduction in stress within 20 minutes after a large imposed strain. Research conducted by Crafco, Inc. and WES [25] indicated similar results with hot-applied sealants. The hot-applied sealants exhibited a significant reduction in stress within 30 to 50 minutes of the imposed strain when tested at room temperature. Some of the hot-applied sealant materials exhibited stress reductions of up to 50 percent even at test temperatures as low as -20°F (-28.9°C).

Time-Temperature Superposition

One of the variables Cook used in the investigation was variations in Shore A hardness of the sealant materials. The sealant materials ranged from a Shore A value of 5 (soft) to 30 (hard). Cook noted that the stiffness of the sealant was temperature dependent. For example, the Shore A value for the soft material would increase to a value of 20 when the temperature of the sealant was dropped to -30°F (-34.4°C). Therefore, the stress relaxation and moduli data obtained at room temperature for the Shore A-20 material would be representative of the material properties of the Shore A-5 material at -30°F (-34.4°C). The ability to extrapolate material properties of sealants at different temperatures based on the data from this study was limited. However, the extrapolation of the data between curves in this study would appear to be one of the first attempts to combine time-temperature superposition principles with pavement joint sealants.

Time-temperature superposition would imply that for amorphous polymers above their Tg, a single empirical function could be used to describe the temperature

dependence of all mechanical relaxation processes. Additionally, the ratio (a_T) of any mechanical relaxation time at one temperature, T, to the mechanical relaxation time at the reference temperature could be derived from static or dynamic viscoelastic tests and the ratio would appear to be similar over a wide range of time [29]. Therefore, the function $a_T(T)$ becomes important in describing the physical properties of a polymeric system. The determination of the ratio a_T will allow a mastercurve to be established by first conducting a series of tests on a material under a given strain rate regime at several different temperatures. The ratio a_T, known as the shift factor, allows test results obtained at high temperatures to be shifted in such a manner so that they represent test results that would have been obtained at slow strain rates with respect to a reference temperature. Conversely, test results obtained at low temperatures could be shifted to represent the test results that would have been obtained higher strain rates with respect to a reference temperature. Figure 2.6 represents a typical mastercurve. All tests were conducted dynamically with strain rate frequencies of 10^{-2} to 10^2 radians/second and at temperatures ranging from approximately -441°F (-263°C) below the reference temperature to -369.4°F (-223°C) above the reference temperature. Test results obtained at temperatures below the reference temperature are toward the right in Figure 2.6 and the test results obtained at temperatures above the reference temperature are shifted to the left. In Figure 2.6, the material property plotted versus frequency is the log of the complex shear modulus (G^*) but the principles of time-temperature superposition are not limited to this property. The horizontal shifting of the individual curves would be accomplished by calculating a shift factor, a_T, using an empirical function such as the Williams-Landel-Ferry (WLF) equation. The WLF equation is [29]:

$$\log a_T = \frac{-c_1 * (T - T_R)}{c_2 + T - T_R} \tag{12}$$

where

$$c_1, c_2 \quad = \quad \text{material constants}$$
$$T \quad = \quad \text{temperature at which the experiment was conducted}$$
$$T_R \quad = \quad \text{reference temperature}$$

One limitation of the WLF equation is that relationship does not necessarily hold true for temperatures that are -279.4°F (-173°C) above Tg. The divergence of the relationship at high temperatures will occur because for most ordinary liquids far above their Tg, the viscosity will become more dependent upon molecular structure [29] causing an increase in variability in viscosity. Supercooled liquids within approximately -279°F (-173°C) of their Tg have highly temperature dependent material properties which will typically independent of the molecular structure. The universal function will also deviate when considering materials at temperatures below Tg. The WLF equation would predict a "monotonic increase of log a_T with decreasing temperature instead of the inflection point which is generally agreed to exist" [29].

Current software programs allow the researcher to specify the reference temperature and then simply move the test results from the other temperature regimes inline with a few keystrokes on the computer. The computer then backcalculates the shift factor and the two material constants. Using the software programs has made developing mastercurves for materials a relatively simple task. Time-temperature superposition has been used successfully in describing the mechanical and electrical relaxation time of many polymeric materials. However, a joint sealant material placed in a joint in the field will also be exposed to various conditions that can cause degradation of the polymer. This would imply that additional considerations must be included to model changes in physical properties caused by degradation.
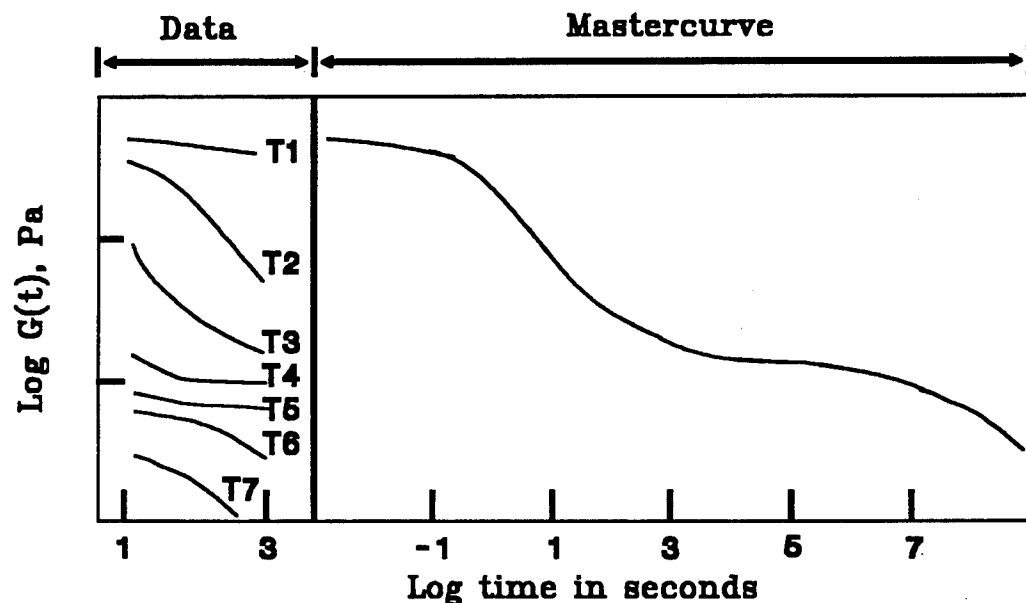
Figure 2.6: Mastercurve Developed Using Time-Temperature Superposition.

Joint Movements

All stresses and strains that occur in the joint sealant material will be caused by movements in the concrete that occur at the joints and cracks. Therefore, to better understand joint sealant performance, it is necessary to briefly discuss the types of movements that occur at the joint and the factors that cause the movement to take place.

There are basically two types of joints used in pavements; longitudinal and transverse. The longitudinal joints are the joints between traffic lanes or between a traffic lane and a paved shoulder. The movements normally associated with longitudinal joints are usually much smaller than those associated with transverse joints. Therefore, the transverse joints will be considered the controlling condition. That is, if the sealant can accommodate the movement in the transverse joints, it will be assumed to be satisfactory for use in the longitudinal joints. The sealing require-

ments between a PCC pavement and an asphalt cement concrete pavement longitudinal joints are unique and will present some additional challenges that are not encountered in transverse joints. These challenges are related to the adhesive and compatibility requirements of the sealant material, and, therefore, are not related to movements that occur in the joints.

Transverse Joints

Transverse joints have typically be divided into three categories; contraction joints, construction joints, and expansion joints. Contraction joints are joints that are sawed or formed into the PCC pavement to relieve longitudinal stresses that develop as the concrete cures. The curing process of PCC involves hydration of the cement paste which in turn will cause a decrease in volume. The volume decrease will create stresses in the concrete which will lead to cracking. By sawing or forming contraction joints in the PCC pavement, the formation of cracks can be controlled. Once the PCC has cured, the contraction joints relieve temperature and moisture induced stresses. The load bearing capability, or the ability to transfer load from one slab to another, of the pavement will be maintained at the joints by some type of load transfer mechanism. The load transfer mechanism can be accomplished through aggregate interlock, or through the addition of load transfer devices such as dowels.

Construction joints will be formed at the end of the work day. The construction joints can be used as either contraction joints or expansion joints if the location of the construction joint coincides with the placement of the planned joint. Construction joints will have a smooth vertical face that extends the full depth of the pavement. Therefore, aggregate interlock cannot be used as a means of load transfer across a construction joint. Load transfer must be accomplished by using some type of load transfer device. If the construction joint did not coincide with a planned joint, tie bars will be used to prevent movement at the construction joint.

Expansion joints are used to relieve expansive stresses that may develop in the PCC. The expansion joints are similar to construction joints in the fact that they are formed full depth in the PCC pavement. The joint width at an expansion joint will typically be 1 inch (25.4 mm) or larger to allow the pavement to expand without

buckling. Because of the large joint width, load transfer devices are required to transfer the load between slabs. In current practice, expansion joints are usually only used adjacent to fixed structures such as bridges. The reduced use of expansion joints from the earlier days of design has occurred because pavement engineers have found that closely spaced contraction joints will provide adequate space for expansion under normal conditions [30]. Since expansion joints are not predominant in current practice and because construction joints can be designed to be non-working joints or contraction joints, joint movement considerations will be focused on contraction type joints.

Horizontal Movements

There are basically two types of joint movement; horizontal and vertical. In most studies concerned with joint movement, the horizontal movements were considered to be caused by temperature and moisture changes and the vertical movements were considered to be caused by induced loads but dependent upon the amount of horizontal movement. Additional factors that influence joint movement will include, slab thickness, joint spacing, type of load transfer device used in the pavement, and the materials used to construct the PCC pavement (i.e., aggregate and cement paste).

The first type of movement that occurs at a joint will result from changes in moisture. Concrete expands with increases in moisture content and contracts with decreases in moisture with the highest moisture content normally being during placement. The curing process of concrete includes hydration of the cement resulting in a reduced moisture content and a decrease in volume. The shrinkage that occurs during hydration, termed autogeneous shrinkage, will be approximately 40×10^{-6} percent at one month after the placement of the PCC pavement up to 100×10^{-6} percent after 5 years. However, the majority of autogeneous shrinkage will occur within the first 3 months after placement of the slabs [31]. Autogeneous shrinkage will be a uniform decrease in volume in unrestrained slabs, and is part of the reason that expansion joints are not required when contraction joints are closely spaced. Not all researchers agree that the early age volume change is due to autogeneous

shrinkage from hydration. The reason for the disagreement is the fact that the PCC is undergoing a complex combination of temperature and moisture changes and these mechanisms may be contributing to the observed volume changes [31]. Regardless of the exact cause of the initial volume change, it does occur. Additionally, the volume change is relatively small and would only be a concern to sealant performance when the joints are first sealed.

Water will also be lost through evaporation, further reducing the moisture content at the pavement surface, increasing the amount of shrinkage. This type of shrinkage is referred to as drying shrinkage. Once the majority of the curing process has completed, remaining moisture changes that occur in the pavement will be seasonal. Many PCC pavements will reach a maximum seasonal moisture content in the winter months which will also the time at which the maximum temperature induced volume decrease will occur. Therefore, in some instances, the increase in volume caused by an increase in moisture may be offset by the volume decrease caused by low temperatures [32]. The extent of offset of the two phenomena will be highly dependent upon the climatic region in which the pavement is located. For example, during the month of January, the average minimum daily temperature in Seattle, WA, would be approximately 33°F (0.6°F). In Miami, FL, the average minimum daily temperature would be approximately 58°F (14.4°C) and in Fargo, ND, the average minimum daily temperature would be approximately 3°F (-16.1°C) [33]. The average monthly rainfall in January for these three locations would be approximately 6, 2, and 0.5 inches (152.4, 50.8, 12.5 mm), respectively [33]. Joint movements from these three locations has not been thoroughly investigated for this comparison, but it is doubtful that the temperature and moisture effects would offset each other in each of these three scenarios. It could be that in the case of Fargo, that the moisture effects would be masked by the temperature affects and in Seattle, moisture effects would be more pronounced.

Typical drying shrinkage coefficients of PCC range from approximately 4.0 to 8.0×10^{-5} inches/inch (10.2 to 20.3×10^{-4} mm/mm). This percentage is significantly larger than the percentages reported for autogeneous shrinkage and, therefore, when

considering the initial sealing of joints, volume changes caused by drying shrinkage will be a greater concern than autogeneous shrinkage. Drying shrinkage will be typically an irreversible process which means that the field performance of a joint sealant used to reseal the joints at some future date would not be affected by the drying shrinkage.

One of the main factors in determining the drying shrinkage coefficient for a particular PCC pavement will be the water/cement ratio. Minimizing the amount of mixing water in the PCC will reduce the drying shrinkage coefficient. Typically for every 1 percent increase in mixing water, the amount of shrinkage will increase by approximately 2 percent. Conversely, the type of cement, cement fineness and composition, and cement content have very little affect on the amount of shrinkage a normal concrete will experience [32]. However, the type of curing procedures used could have a significant effect on the amount of shrinkage or size of volume changes that the PCC slab will undergo.

The above discussion considers instantaneous moisture loss. Realistically, the moisture loss will begin at the pavement surface creating a moisture gradient in the pavement slab. Conversely, when the pavement absorbs water, this will also begin at the pavement surface. These two conditions cause the pavement slab to curl [34]. If pavement surface was drier than the bottom of the slab, then the slab will curl upward. If the bottom of the slab was drier, the slab will curl downward. Typical ranges for the deflection that could occur at the joint were difficult to determine because additional factors such as the weight of the slab and the use of load transfer devices will greatly affect the amount of movement at the joint. The amount of joint movement will also be dependent upon which portion of the slab was being considered. For example, along the joint, the slab corners will normally exhibit larger movement than the center of the slab. Movement at the joint will also not necessarily be uniform between the two adjacent slabs due to differences between the slabs. Additionally, research has indicated that in some Illinois PCC pavements, significant drying only occurred in the top 2 inches of the pavement surface [35]. Field and laboratory tests indicated that the level of saturation below the top 2

inches remained at 80 percent or more throughout most of the year. The tensile stresses that developed in the top 2 inches were shown to exceed the tensile strength of the PCC pavement. The microcracks that developed from the tensile failure at the pavement surface reduced the total amount of strain expected at the joint. But curling stresses will be present in most pavements with the pavement surface in tension and the bottom in compression. Therefore, curling will affect the movement at the joint and should be considered when evaluating sealant performance.

Changes in temperature can affect joint movement in two ways. The PCC expands as the temperature increases and contracts as the temperature decreases. The linear coefficient of thermal expansion for PCC ranges from approximately 3.2 to 7.0 x 10^{-6} inches/°F ([32]. The maximum or minimum joint opening can be calculated by [36]:

$$dL = 12 * C * L * (\alpha * dT + \varepsilon) \tag{13}$$

where

dL	=	joint opening (in inches)
α	=	linear thermal coefficient of expansion/contraction
ε	=	coefficient of drying shrinkage (0.50 to 2.50 x 10^{-4} inch/inch (12.7 to 63.5 x 10^{-4} mm/mm) for new concrete, 0 for old concrete)
L	=	joint spacing (in feet)
dT	=	temperature range (T - T_{min}) or (T_{max} - T), where T is the temperature of the pavement at the time of sealing, T_{min} is the minimum pavement temperature and T_{max} is the maximum pavement temperature.
C	=	adjustment factor due to friction restraint between the slab and subbase (0.65 for stabilized subbase, 0.80 for granular subbase and 1.0 if the slab is placed on the subgrade.

Although ε is called the coefficient of drying shrinkage, this term is used to account for the initial volume change and not seasonal drying shrinkage. This conjecture was based upon the fact that the U.S. Air Force Manual [36] equated the coefficient to 0 for older pavements. The above equation could be used by designers to determine the appropriate slab length by substituting in 0.04 inches (1 mm) in for the joint opening, dL, the expected total temperature differential, dT, and calculating the corresponding slab length, L. A joint opening of 0.04 inches (1 mm) was used to determine slab length because a joint opening of this magnitude or less has been accepted as an adequate joint opening for undoweled plain jointed concrete. Adequate in this case means that the ride quality of the pavement will be satisfactory and sufficient aggregate interlock could be maintained to transfer load from one slab to the next. However, some studies which were summarized in the Washington State DOT (WSDOT) pavement Guide [2] indicate that a joint opening of 0.035 inches (0.89 mm) or less was required to provide satisfactory interlock.

Vertical Movements

Temperature gradients could also cause PCC pavements to warp which would be very similar to the curling phenomena caused by moisture gradients. If the temperature on the pavement surface was greater than the temperature at the bottom of the slab (as often occurs during the day) then the slab warps downward. At night the temperature gradient would be reversed and the slab warps upward forming a bowl. As in the case of curling, typical deflection ranges that occur at the joint because of warping were difficult to provide. The difficulty arises from the fact that aggregate interlock between slabs and the weight of the slab will affect the amount of deflection. The deflection will be further hindered if load transfer devices were used in the joints. Therefore, field measurements are normally required to accurately determine vertical movements caused by temperature gradients. Temperature induced vertical and horizontal movements have been made for some pavements in Florida [37]. The measurements were made on two adjacent slabs from January to June, 1986, excluding March. Vertical measurements for the two slabs ranged from 0.046 to 0.098 inches (1.2 to 2.5 mm) at the corners with the

average displacement for the five months at slab 3 being 0.072 inches (1.8 mm) and 0.063 inches (1.6 mm) for slab 4. The measurements indicated that the corner displacements were greater than at other portions of the slab and that the displacements between the slabs were not uniform. Horizontal movements were made on both doweled and undoweled joints. The measurements were made during the same five months with the monthly averages ranging from 0.030 to 0.043 inches (0.8 to 1.1 mm) for the undoweled slabs and 0.009 to 0.023 inches (0.2 to 0.6 mm) for the doweled joints. From these measurements, it would appear that temperature induced vertical movements are more significant than the temperature induced horizontal movements. It will therefore be necessary to consider both horizontal and vertical movements induced by temperature when considering the field performance of sealants.

Usually when pavement engineers discuss vertical joint movements they are referring mainly to load induced joint movements instead of movements caused by curling or warping. Load induced joint movements occur when the slab is exposed to traffic. The amount of movement that occurs will depend on the applied load, the rate of loading, the support supplied to the slab by the subbase, the joint opening at the time of loading, and whether or not load transfer devices have been used in the joint. The relationship between each of these factors will be complex. As a result of this complexity, a typical range of vertical movement cannot be provided. However, vertical movement in a joint will normally be greater in older pavements versus newer pavements and in pavements without load transfer devices versus pavements that include load transfer devices. Although a typical range cannot be given, the results of the Zero-Maintenance Design study indicated that the critical level of faulting was 0.20 inches (5.1 mm). WSDOT considers faulting of 0.125 to 0.25 inches (3.2 to 6.4 mm) to be low severity while the Pavement Condition Index (PCI) developed by the U.S. Army Corps of Engineers considers low severity faulting to be 0.125 to 0.375 inches (3.2 to 9.5 mm) [38]. The amount of faulting agencies use to indicate when a pavement requires repair varies. However, when faulting reaches 0.20 to 0.25 inches (5.1 to 6.4 mm), the ride quality of the pavement would be less

than satisfactory for small vehicle traffic and would be severe for unloaded truck traffic. The maximum amount of faulting that an agency would allow before conducting maintenance will be a concern when considering joint sealant field performance because this movement could be significantly larger than some of the types of joint movement.

A study conducted in Massachusetts in 1964, [39] indicated that in the case of expansion joints, the vertical deflections at the joint were measured to be 5 percent of the horizontal strain. Therefore, the study concluded that vertical movement in expansion joints did not seem to be important when compared to horizontal movement in the performance of joint sealants. The study also concluded that field measurements indicated that temperature effects were the most prominent cause of horizontal movements in "sound" concrete. The Massachusetts study did not make any generalizations concerning the vertical movement at contraction joints. From 1972 to approximately 1984 [40], a study was conducted in Ohio to determine the actual magnitude of vertical movement. In this study, the deflections at the joint were induced by a truck with a known axle weight. Additionally, potential variances caused by warping were considered by taking measurements both in the morning and in the afternoon. The study verified that the type of subbase, the amount of the load, the speed of the load across the joint, the location of the load (edge of slab versus center), and time of day that the measurement is taken will affect the magnitude of the vertical movement. The study also concluded that the vertical movements measured at the joints were relatively small when absolute values (5.98 to 8.46×10^{-3} inches (0.15 to 0.21 mm)) were considered. Therefore, potential fatigue failure in the concrete slab was considered to be a more significant concern than the vertical movement.

When evaluating the sealant material in the joint, it may be possible that the vertical movement will be more critical especially during winter months when the joint is at its maximum opening. The horizontal movement that will occur as the temperature or moisture content of the slab changes will be gradual whereas the vertical movement induced by traffic will be rapid. The sealant material should

exhibit a viscous type behavior at the slower strain rate and exhibit a more elastic behavior at the fast strain rate.

The best method to determine the joint movements of a particular pavement would be to physically measure them. Typically, the best method of determining the field performance of a material would be to place it in the field and monitor the performance or to simulate field conditions and test the materials in the laboratory. The Swedish National Testing Institute conducted a research effort [41] in which the field conditions were simulated in the laboratory. Temperature changes, joint or crack movement, rate of joint movement, and rainfall were monitored in various locations throughout Sweden. A testing device was then developed to replicate the monitored environmental conditions. Sealant materials were then tested using the expected field conditions and the best performing material would be selected for use. The drawback to this type of testing was the length of time required to test the material. One cycle in the environmental testing equipment took approximately 35 to 40 hours. The cycling was in addition to any preconditioning of the sealant which could take up to 12 weeks. Therefore, to complete field simulation testing of a sealant material could take over 15 weeks. For user agencies that normally must accept the low bid for a project and do not have the advantage of developing qualified products lists, a 15 week turn around time for sealant testing would be unrealistic. A combination of material characterization tests and finite element modeling would appear to be the solution.

<p align="center">*Pavement Stress Considerations*</p>

Stresses are developed in the PCC pavement slab by each of the mechanisms that have been described above. Normally, pavement engineers have been more concerned with the stresses that develop in the pavement instead of the strains that occur due to these stresses. The emphasis on stresses in the pavement stems from the fact that a build up of stresses in the pavement could lead to cracking which in turn would reduce the durability of the pavement structure. However, when considering the field performance of joint sealant materials, the focus shifts to the strains at the joint induced by the stresses. Virtually the only time that stresses in

the PCC slab become important to joint sealant performance would be when the stresses in sealant become greater than the ultimate tensile stress of the PCC.

The tensile strength in PCC can be assumed to be approximately 10 percent of the compressive strength of the concrete. Compressive strength of PCC is dependent upon numerous variables, two of which are the materials and proportions of the materials used to produce the concrete, and the procedures used to cure the PCC. However, typical compressive strengths for standard concrete range from approximately 3,500 to 6,500 psi (24.1 to 44.8 MPa) [34]. This would imply that the typical range for tensile strength would be 350 to 650 psi (2.4 to 4.5 MPa) and for a joint sealant material to cause spalling, the tensile stresses developed at the sealant/concrete interface must exceed 350 to 650 psi (2.4 to 4.5 MPa).

Cook [12] stated that spalling failures in PCC joint should not be attributed to sealant materials. Cook continued the argument by stating that the spalling failures were caused by "poor concrete, not a strong sealant." Cook based these statements on the low values of stress exhibited by the polysulfide sealant when tested at room temperature. However, tests conducted at WES [6] indicated that when some sealants were exposed to low temperatures (-20°F (-28.9°C)) and high strain rates (2 inches/minute (50.8 mm/minute)), stresses exceeding the tensile strength of PCC could be developed. Some of the excessive stresses were developed within 4 to 10 percent elongation which corresponds to a strain of approximately 0.02 to 0.05 inches (0.5 to 1.3 mm). The test conditions and the amount of strain at failure could represent a pavement with minor faulting during winter in the northern United States. The excessive stress build up was only noticed with some of the hot-applied sealants and one or two of the cold-applied, two-component sealants. The excessive stress buildup was not exhibited by the low modulus silicones and will therefore not be considered as a potential mode of sealant failure. Any spalling type failures that occur in which the PCC remains adhered to the silicone sealant will be normally attributed to poor quality PCC.

Many computer programs have been developed to model the movements and stresses that occur in PCC and many studies have been conducted to compare the

advantages and disadvantages of several of these programs [42, 43]. Each of the programs have advantages and disadvantages when modeling certain types of conditions in the pavement and some programs are more user friendly than others. However, the strains predicted by these models could be used in numerical sealant models to depict specific site conditions if measurements are not available.

<u>Expected Precision</u>

The coefficients of variation from the test results of the material characterization of the silicone joint sealants were expected to be in the 20 to 30 percent range. This particular range may seem too large for data that are to be used in numerical analysis; however, this range is generally considered satisfactory for material characterization techniques used by the pavement industry. For example, the approximate coefficient of variation for asphalt cement characterization using the dynamic shear rheometer was approximately 34 percent [44]. The seemingly high coefficients of variation generally obtained during civil engineering material characterization testing can be attributed to the non-uniformity of the material being tested and the small sample population used to characterize the material.

The current joint sealant specification tests used for hot-applied joint sealant materials exhibited a wide range of coefficients of variation. A recent study conducted jointly by WES and Crafco, Inc., [25] indicated that the coefficient of variation for the unaged initial indentation test (a test procedure similar to a penetration test) ranged from approximately 9 to 64 percent. The coefficient of variation for the same test on aged joint sealant materials ranged from 0 to 69 percent. The coefficients of variation for the other tests such as flow, penetration, and resilience ranged from approximately 1 to 177 percent. The potential reasons of the large variation given for these test procedures were variability of the sealant materials and variability of the test procedures. Results of the WES/Crafco study delineated the need for round robin testing to allow precision and bias statements to be developed for the ASTM sealant specifications. The round robin testing indicated the variation exhibited in the test methods were due in part to the type of material being tested. For example, the results of two properly conducted

penetration tests in different laboratories of a hot-applied material which exhibited a penetration between 50 and 70 should not differ by more than 15 penetration units. The results of two properly conducted penetration tests in different laboratories of a hot-applied material which exhibited a penetration between 71 and 85 should not differ by more than 48 penetration units. The penetration requirement for Federal Specification SS-S-1401C is a maximum of 9 mm or 90 penetration units. The variation would imply that a material which in fact complied with material specification requirements could yield test results which indicated non-compliance. The single operator precision for resilience was 3 units and the multi-laboratory precision was 33 units, again indicating that a material that actually conformed to specification requirements may be reported in non-compliance. An industry accepted specification did not exist for silicone sealants; therefore, precision and bias data were not available for the procedures used to test silicone sealants. However, variability similar to that exhibited in the other types of sealant specifications would be expected.

Another area of civil engineering material characterization that had exhibited a great deal of variability was soils testing. In the 1960's, a round robin was conducted which included approximately 100 of the "top" testing laboratories [45]. Three different soil materials; a low plasticity Vicksburg loess, a medium plasticity lean clay, and a high plasticity buckshot clay, were sent to the testing laboratories. The tests conducted on these soils were liquid limit, plastic limit, grain size, specific gravity, and standard and modified moisture-density relations. The basic conclusion of the study was that the variability of the routine tests was "...of considerable magnitude in many instances." The variation in testing was highlighted by the fact that the 95 percent confident region for the specific gravity of the high plasticity buckshot clay ranged from a minimum of approximately 2.4 to a maximum of approximately 2.9. This specific gravity range included almost every type of soil that had ever been evaluated.

Another material characterization technique was aggregate sieve analysis. In this procedure, an aggregate sample was obtained and separated based on particle

size using a series of sieves. One specification typically used for conducting a sieve analysis is ASTM C 136. The precision estimate for the coarse aggregate analysis listed in the precision and bias statement of the specification indicated that the coefficient of variation ranged from approximately 30 to 35 percent. The variation was attributed to sampling error, either a non-representative sample or an overcharging of the sieves, or equipment and procedure errors, i.e., the condition of the equipment or the duration of aggregate sieving [46].

This brief review indicates that the variability of the sealant characterization may be larger than would normally be desired for numerical analysis. However, if the coefficients of variation for the characterization technique are less than approximately 20 to 30 percent, then a great advancement would be made in joint sealant characterization.

CHAPTER 3 - EXPERIMENTAL

Previous researchers such as Tons [39] and Wang [47] have demonstrated that finite element analysis can be used to model silicone pavement joint sealants and there are a number of finite element analysis programs commercially available. Therefore, the development of a new computer code to model the sealant behavior was not needed. Consequently, the major emphasis of this research was to develop an appropriate material characterization methodology.

The investigations conducted by Tons [39] and Wang [47] assumed that the material properties of the silicone sealants were not sensitive to temperature or aging. These assumptions were valid for first approximations and they allowed the material evaluation techniques to be simplified. However, if characterization techniques were to be advanced to sealants that exhibit higher temperature and aging sensitivity, then the dependence upon these factors must be determined.

The approach developed to achieve the stated objective of determining a material characterization methodology that could be used in addition to current sealant specifications or as a basis for a new sealant material specification investigated several variables. The first variable was curing. Any new methodology must be responsive to user, manufacturer, and contractor needs. One complaint that all of the above groups had was that current silicone specifications require several weeks to complete thus potentially delaying the completion of a project. Therefore, it was important that methods of accelerating the cure be examined to minimize the length of testing. The next important parameter to consider when developing a potential requirement for specification application was to investigate how the material properties would change as the material weathered and aged. Silicone sealants are typically known for their resistance to weathering, and, therefore, many researchers have eliminated the weathering or aging characteristics of the silicone from investigation. However, differences should exist between different silicone materials and if the methodology was to be used on other types of sealants, aging characteristics need to be considered. The most important item of the material characterization methodology was the selection of the procedure used to actually test the material. Usually it would be desirable to conduct two or three tests that will

fully characterize a material instead of conducting one single test and basing all conclusions on that one test. It would also be important that the tests selected tests take into account the viscoelastic nature of the material and provide fundamental physical properties of the material being tested. All of these items were considered as the test plan was developed.

Dynamic Shear Rheology (DSR)

Numerous small strain experiments are available for rheological analysis. Stress relaxation, creep and sinusoidal oscillations are some of the more common types of tests that are conducted. The dynamic shear rheometer is capable of performing these three common tests and with data reduction and manipulation software such as the Innovative Rheological Interface Software (IRIS) [48], the rheological properties of a material can be modeled by conducting only one of the tests. The initial modeled results must be verified to ensure the IRIS models are valid for the specific material being tested, but once verified the total amount of testing can be reduced.

DSR was developed to investigate polymeric systems but has been adopted by other disciplines as a means to characterize the properties of materials for performance based specifications. One example of the growing acceptance of DSR was it's inclusion in the Strategic Highway Research Program (SHRP) asphalt binder specification [49]. The DSR procedures in that specification allowed the stiffness of the asphalt binder to be determined at temperatures representative of field conditions. Previous asphalt binder specifications measured the viscosity of the asphalt binders at high temperatures (typically 140°F and 275°F (60°C and 135°C)) and/or the penetration at 77°F (25°C).

One of the greatest potentials of the DSR was it's ability to characterize the properties of a material over a wide range of temperatures and frequencies or rates of loading. This characterization was achieved through time-temperature superposition as discussed in Chapter 2. The DSR was selected for this research investigation based on it's ability to characterize material properties over a wide

range of conditions, sample preparation was relatively easy, and the same tests can be conducted on field obtained samples and laboratory prepared samples. Therefore, models developed on laboratory prepared and conditioned samples can be used to potentially determine the remaining useful life of an in-place material by testing samples taken from the field.

The mode of testing that was selected for DSR testing was sinusoidal oscillation using parallel plates. The sinusoidal mode was selected because the samples were soft solids. Other testing modes such as continuous shear techniques were more suited to liquid materials and were not as applicable to solids. In addition, sample preparation was simple and the samples were easier to load and unload from the testing equipment. However, the use of parallel plates did introduce some potential errors into the experiment. Typical errors that could occur when using parallel plates are inertia and secondary flow, edge effects, shear heating, and a nonhomogeneous strain field.

DSR Testing Concepts

Before discussing the types of errors that can occur with the sinusoidal oscillatory testing using parallel plates, the testing concepts of the DSR must be understood. When using sinusoidal oscillation, a sinusoidal strain will be placed on the sample. Within the first few cycles, the stress response will also begin to oscillate sinusoidally at the same frequency as the strain but the stress is typically shifted by some phase angle δ. The correlation between the stress and strain can be mathematically represented by [50]:

$$\gamma = \gamma^{o} \sin \omega t \tag{14}$$

$$\tau(t) = \tau_{o} \sin(\omega t + \delta) \tag{15}$$

where

$$\gamma \quad = \quad \text{sinusoidal shear strain}$$

$$\tau(t) \quad = \quad \text{time-dependent shear stress}$$

τ_0 = peak shear stress or amplitude

ω = radian frequency (2π times the cycles per second)

δ = phase angle or lag between the applied strain and the resulting stress

The stress response can be separated into two components of the same frequency; one in phase with the strain input or wave ($\sin \omega t$) and one $90°$ out of phase with the strain wave ($\cos \omega t$). Using the in phase and out of phase components, equation 15 would become:

$$\tau(t) = \tau_o' \sin \omega t + \tau_o'' \cos \omega t \tag{16}$$

From equation 16 it follows that:

$$\tan \delta = \frac{\tau_o''}{\tau_o'} \tag{17}$$

Two dynamic moduli are suggested from the stress components; the elastic or storage modulus G' and the viscous or loss modulus G''.

$$G' = \frac{\tau_o'}{\gamma_o} \tag{18}$$

$$G'' = \frac{\tau_o''}{\gamma_o} \tag{19}$$

The $'$ and $''$ notation in the above equations represent the fact that imaginary numbers were introduced in the description through $e^{i\theta} = \cos \theta + i\sin\theta$ where i is equal to $-1^{1/2}$. From this the complex modulus G^* can be defined such that:

$$\tau_o = |G^*|\gamma_o \tag{20}$$

where

$$G^* = G' + iG'' \tag{21}$$

However, G'' can be physically measured because it will be a measure of the energy dissipated per cycle of deformation per unit volume [50]. Therefore, the magnitude of the complex modulus can be reported as:

$$|G^*| = (G'^2 + G''^2)^{1/2} \tag{22}$$

The complex moduli reported in this research were derived from the above equation.

A controlled strain rheometer was used for the silicone sealant characterization in this study. The controlled strain rheometer in the oscillatory mode measures the magnitude of G^* and the phase angle or lag between the imposed strain and measured stress. Using these measurements, software was used to decompose the data into G' and G''. The G' and G'' data along with the phase angle δ were transferred to the IRIS software which was used to develop the mastercurves at the selected temperatures. The mastercurve data was then used to calculate the discrete stress relaxation spectra which converted the data from the frequency domain to the time domain. The conversion between the two domains was made through a method called "Parsimonious Modelling" because it attempted to find the spectrum with the smallest number Maxwell modes which still adequately represented the data [51]. The output of the IRIS discrete relaxation spectrum was a series of weighted factors with the corresponding relaxation times. These values were then be used to calculate the stress relaxation modulus, G(t) using the following equation:

$$G(t) = \sum_{i=1}^{N} g_i e^{-t/\lambda_i} \tag{23}$$

where

$$N \quad = \quad \text{number of modes}$$

$$t \quad = \quad \text{time of interest}$$

$$\lambda_i \quad = \quad \text{relaxation time}$$

$$g_i \quad = \quad \text{weighted factor corresponding to the relaxation time}$$

The quality of the fit of the parsimonious model was dependent upon the number of relaxation modes per decade of the dynamic data. Obviously the fit would become poor if too few modes were used and the quality of the fit would increase as the number of modes increased. Winter, et al [51] found that 1.4 to 1.7 modes per decade were the optimum range for fitting the data. Above this range, the fit did not significantly improve; however, the exact optimum range for the number of modes was material dependent.

Potential Testing Errors

The potential problems with the parallel plate geometry as listed above are inertia and secondary flow, edge effects, shear heating, and a nonuniform strain field. Inertia and secondary flow are generally considered to be more of a problem when testing fluid materials rather than solids and much of the analysis of inertia and secondary flow has occurred using the cone and plate geometry but the results of inertia or secondary flow will be similar when using parallel plates [50]. Inertia forces tend to pull the plates together creating a negative normal force. The inertia forces create secondary flow in the sample for large cone angles which increases torque leading to erroneous results.

Edge effect failures can be identified by the separation of the free surface of the material from the plates. Edge failures become more predominate in high viscosity materials like polymer melts. When a sample experiences an edge failure, the torque and normal force values will drop creating erroneous results. Edge failures can significantly limit the shear rate range that can be effectively used to characterize polymers.

One other type of edge effect that could be present when conducting experiments would be hardening or a changing of the sample at the gas-liquid interface. A nonvolatile oil could be applied to the edge of the sample to minimize the changes that may occur. Since the silicone sealants used in the characterization investigation were cured before analysis, hardening or changing at the free edge was not expected.

Shear heating would imply that the temperature of the sample increased to some maximum temperature above the isothermal temperature of the test. The effect that the rise in temperature will have on the test results are dependent upon the magnitude of the temperature rise and the temperature sensitivity of the material being tested.

One method to correct for each of the three problems; inertia and secondary flow, edge effects, and shear heating would be to reduce the gap opening used for the tests. When testing a liquid material, adjustments in the gap opening would be easy to make. When testing a solid, the gap opening will be determined by the thickness of the sample being tested. Therefore, gap opening adjustments must be made by reducing the thickness of the sample instead of simply lowering or raising the plates. If the plates were adjusted without adjusting the sample thickness, the plates would either compress the sample creating normal forces in the sample or the plates would not be in adequate contact with the sample causing slippage between the plates and the sample. One potential problem with using small gap openings would be the fact that any misalignment between the plates becomes more critical at the small openings.

The strain field on the sample was not homogeneous and was dependent upon the distance from the center point of the sample or radius with values ranging from zero at the center of the sample to a maximum at the outermost edge. Therefore, the results obtained from testing using the parallel plate configuration were apparent values. The controlled strain rheometer software included correction factors that were sample configuration dependent, i.e., one set of factors for a 0.47 inches (12

66

mm) diameter parallel plate configuration, another set for 0.98 (25 mm) diameter parallel plates, etc. These factors allowed the material values obtained using one set of plates to be compared with the results obtained using a smaller or larger diameter plate, i.e., directly compared material properties.

The importance of sample thickness and gap opening has been previously discussed, but there were additional factors that must be considered based upon the equipment being used to conduct the rheological analysis. For example, to obtain accurate results using the controlled strain rheometer, the measured stress must be above five percent of the range of the transducer and the correction factor used to account for "play" in the equipment as calculated by the equipment software must be less than 0.2. Conversations with technical representatives from rheometer manufacturer indicated that measurements that occurred in the one percent range of the transducer were usually satisfactory. All measurements that were recorded that were not one percent or greater in range and/or had a correction factor greater than 0.2 were discarded.

Linear Viscoelastic Region Determination

The first step in conducting DSR testing was to determine the linear viscoelastic region of the material. The majority of equations that were used in the modeling assumed the material was being analyzed in the linear viscoelastic region or the region where G(t) did not vary with strain. The linear viscoelastic region of the silicone sealants was determined by conducting a strain sweep at -22 and 86°F (-30 and 30°C). The oscillation frequency used to conduct the strain sweeps was 1 Hz. The two strain sweeps were examined and the amplitude for testing was selected in the linear viscoelastic region where the range of the equipment was above one percent and the correction factor was less than 0.2. In most cases, the silicone sealant was linear from an amplitude of 10 to 80 percent. This indicated that the sealant could be tested using any of these amplitudes. This fact became more important when the range of the transducer dropped below one percent as frequencies below 1 Hz were used. In these cases, the test would be terminated, the

amplitude increased, and the test conducted again. If the range of the equipment remained below one percent over the entire linear viscoelastic region, the data were deleted from the analysis.

Two different plate diameters were used for the initial strain sweeps, 0.47 inches (12 mm) and 0.98 inches (25 mm). The majority of strain sweeps conducted using the 0.47 inches (12 mm) parallel plates indicated that regardless of amplitude, the range was always below one percent. This was not a problem for laboratory prepared samples because they could be molded or punched into the desired diameter. Field samples, however, would have to be cut from material that normally would be approximately 0.24 to 0.32 inches (6 to 8 mm) deep and 0.24 to 0.51 inches (6 to 13 mm) wide. The size restriction comes from the fact that most joints are 0.24 to 0.51 inches (6 to 13 mm) wide and sealants are typically installed to have a depth of approximately 0.24 to 0.32 inches (6 to 8 mm). Therefore, it may not be possible to obtain samples from the field that will provide adequate characterization unless hardening or aging of the sealant has occurred.

Once the linear viscoelastic region had been defined, another sample was placed in the rheometer to conduct the rheological testing and the selected temperature was programmed into the computer controls. After a strain sweep had been conducted on a sample, it was possible that the linear viscoelastic region had been exceeded. If the linear viscoelastic region had been exceeded, the material properties of the sample were permanently changed. The determination of whether or not the linear viscoelastic region had been exceeded could be made by evaluating the strain sweep data. If the data exhibited a 5 percent or more decrease in the shear modulus, the point at which the modulus decreased would be considered the limit of the linear viscoelastic region. Subsequent tests on a material that had been tested beyond the linear viscoelastic region would result in lower modulus values.

DSR Testing Procedure

The test temperatures used for the rheological testing were -22, -4, 14, 32, 50, 68, 86, 104, 122°F (-30, -20, -10, 0, 10, 20, 30, 40, and 50°C). The sample was placed into the equipment, the upper plate was lowered onto the sample and the temperature set at -22°F (-30°C). After the environmental chamber equilibrated at the selected temperature, an additional five minutes were allowed for the sample to equilibrate at the test temperature. Five minutes were determined to be sufficient by placing a small thermistor embedded in silicone between the parallel plates in the environmental chamber. The temperature was set at -22°F (-30°C) and the temperature of the thermistor was recorded. It was found that within two to three minutes after the environmental chamber equilibrated, the temperature of the thermistor reached the temperature of the chamber. An additional two minutes were added to this time to ensure the entire sample had reached test temperature. The temperature probes of the DSR were also calibrated using the thermistor and glass thermometers manufactured to meet the requirements of ASTM E 1. The upper plate was then lowered to produce a preload equivalent to 0.11 pounds (50 grams) to ensure the plates were in adequate contact with the sample. This procedure also allowed the gap opening to be adjusted to account for the thermal contraction of the silicone. Four measurements were made at each test frequency (0.02, 0.04, 0.06, 0.08, 0.1, 0.2, 0.4, 0.6, 0.8, 1, 2, 4, 6, 8, and 10 Hz). The first test was discarded and the last three were averaged in an effort to provide a more representative measurement. After completion of the -22°F (-30°C) testing, the test temperature was programmed to -4°F (-20°C) and the steps repeated. The 122°F (50°C) data were the last data collected for the samples. A second sample of the same material was placed in the rheometer and the procedures repeated. The second sample allowed the variation between samples to be evaluated. The three recorded measurements at each temperature of the first sample were averaged with the corresponding three measurements of the second sample. The averaged data were converted into the

IRIS format and used to develop the mastercurves and corresponding stress relaxation spectra.

Variability Affecting Test Results

Three additional variables were evaluated before the analysis procedures were finalized. The first variable concerned the preparation of the samples, more specifically, the punching out of samples from a cured sheet of material versus molding the samples to the desired diameter. The punching out of samples could have created residual stresses in the samples that would have altered the measured properties. Two of the silicone sealants were cast into 0.98 and 0.47 inches (25 and 12 mm) diameter molds that had a depth of approximately 0.06 inches (1.5 mm) and 0.1 inches (2.5 mm). The same two sealants were also cast into molds that were approximately 2.95 inches (75 mm) wide by 2.95 inches (75 mm) long by 0.06 or 0.1 inches (1.5 or 2.5 mm) deep. Evaluation of these results indicated that no measured differences could be detected between the cast and punched samples. The coefficients of variation between the G' of the cast samples and the G' of the punched samples was less than 10 percent and in many instances, it was less than five percent. The low coefficients of variation was only true for the samples that were 0.06 inches (1.5 mm) thick. The data indicated that the samples could be punched from a cured sheet of material without greatly affecting the measured results. It also implied that the material properties of sealants used in the field should not be altered by cutting 0.06 inches (1.5 mm) slices of material and then punching a sample from the material slice.

The second variable examined was sample thickness. From the comparison of cast versus punched samples, it became evident that samples that were greater than approximately 0.08 inches (2 mm) exhibited an increase in variability. Additional samples were produced which were less than 0.04 inches (1 mm) thick. These samples also exhibited greater variability than the 0.06 inches (1.5 mm) which was potentially caused by small misalignments of the parallel plates. This data indicated that samples should be cast to a thickness of approximately 0.06 inches (1.5

mm.) It also implied that any samples cut from field samples must also have a thickness of approximately 0.06 inches (1.5 mm).

The last variable examined was the ordering of the temperature during the testing. One sample was tested using a temperature ordering of -22 to 122°F (-30 to 50°C) in increments of approximately 18°F (10°C). A second sample was tested using a temperature ordering of -22, 122, -4, 86, 14, 68, 32, and 50°F (-30, 50, -20, 30, -10, 20, 0, and 10°C). The results from this testing indicated that the ordering of the temperature did not affect the measured results as long as the sample thickness was approximately 0.06 inches (1.5 mm) at 86°F (30°C) and the range and correction factor requirements of the equipment were met.

<u>Accelerated Curing</u>

Silicone sealant manufacturers have indicated that one method that could be used to accelerate the cure time would be to simply add a small percentage of water to the silicone sealant. By accelerating the curing process, the silicone sealant could be tested within 2 to 3 days instead of several weeks. However, some problems have been noted when accelerated curing was used in the testing of building sealants. Panek [52] noted that accelerated curing and heat aging could enhance the performance of the material being tested. For example, a material that was cured at an elevated temperature could exhibit better adhesion properties than a sealant cured at room temperature. The increase in adhesion could be the difference between specification conformance and non-conformance. The accelerated curing that was proposed for this material characterization investigation included the addition of water to the sealant or an increase of temperature and humidity. Consequently, it was necessary to compare accelerated cure sample properties to normally cured sample properties to verify that the material properties were not altered.

The original idea for accelerated curing for this project was obtained from one of the silicone joint sealant manufacturers. The manufacturer used continuous shear rheology to monitor the curing of the silicone sealant to determine if it would

conform to material specifications. The manufacturer had developed correlations between the curing of one of their sealants and the specification requirements. To accelerate the cure of the silicone material for this testing, the manufacturer added two percent by weight of water to the silicone sample. The addition of the water allowed the testing to be conducted in a matter of hours as opposed to days if the water had not been added.

The methodology desired for material characterization for potential inclusion in a material specification would be to test samples that had already been cured instead of testing the material as it cured. This would allow samples taken from the field to be tested using the same procedures conducted on the original laboratory samples. The testing of the field samples was desirable for two reasons; first there are no industry accepted procedures for testing field samples and secondly, the testing of the field samples would help refine field performance predictions models. Therefore, the accelerated curing procedures were modified slightly from the manufacturers procedure so that the samples could be molded into sheets. Two percent by weight of water was added to the silicone, the silicone was poured into a mold and allowed to cure. The silicone was cured enough to prepare DSR samples, but the mixing process created voids in the cured sealant. The voids created non-uniformity in the samples which would adversely affect the results obtained from the DSR. Additional samples were prepared by adding water and then placing the samples under a vacuum. These samples also had voids in them and were not satisfactory for DSR testing.

The second method investigated for accelerating the curing process consisted of pouring the silicone sealant into a mold and placing the mold on a 2 inch (50.8 mm) stand in a one gallon (3.79 liters) container. Water was poured into the container to a depth of approximately 1 inch (25.4 mm) and a lid was loosely fitted on the container. The container was then placed in a forced-draft oven set at 158°F (70°C) for 24, 48, or 72 hours. The addition of heat to the accelerated curing process raised some concern that the curing mechanisms and physical properties could be

altered. Therefore, control groups were laboratory cured for 21 days for comparative analysis.

Aging Characteristic Analysis

Silicone sealants are generally considered to be resistant to weathering and aging effects, but some testing conducted by WES indicated that silicone sealants manufactured by different manufacturers exhibited some aging and the aging varied by manufacturer and product. Typical equipment used in the past to investigate aging characteristics have been twin carbon-arc or QUV weatherometers and forced-draft ovens. While some of the investigations indicated that the sealants did exhibit physical property changes, these changes often occurred only after 1000 to 2000 hours of conditioning.

One potential aging technique was the pressure aging vessel (PAV). This technique involved placing a sample into a pressure vessel, heating the vessel to 194 or 212°F (90 or 100°C) depending upon the material being tested, and pressurizing the vessel to 300 psi (2.07 MPa). The SHRP procedure conditioned the asphalt cement samples for 20 hours at the selected conditions. The silicone sealant samples were conditioned for 24 and 72 hours before DSR testing. One potential problem with this aging technique was that the materials in the accelerated cured condition may not have been completely cured. The increased pressure on the sample could force air into the material. When the pressure was released on the PAV, any air that may have been forced into the sample would rapidly leave the sample and could cause voids to form in the sealant material. Any voids formed in the sealant sample would adversely affect the sample during DSR testing.

A second method of accelerated aging was also conducted on selected silicone sealant samples. The second method consisted of placing the silicone samples in a forced-draft oven set at 200°F (93°C) for 7 or 14 days. This conditioning technique was selected because it is a common aging technique used for other types of field-molded joint sealants. Since the adhesive characteristics of the sealant material were

not being investigated, the concerns associated with the adhesion properties of the sealant being increased due to the conditioning were not relevant.

<u>Data File Conversion</u>

Before describing the results obtained from the DSR characterization, it is necessary to describe how the data files were generated by the DSR and then analyzed and converted to provide the mastercurves, the discrete relaxation spectra, and ultimately the finite element model. The raw data collected on each sample included the test temperature, the storage modulus (G'), the loss modulus (G''), the phase angle (δ), the frequency in Hz, data correction factor, amplitude, and range for each measurement. This information was stored in a *.ost data file.

Four measurements were made at each frequency and two tests were conducted at each condition. Therefore, a total of eight measurements were made on each sealant material at each condition. The first measurement of each *.ost file was eliminated and the remaining six (three from each test) were averaged, and the standard deviation and coefficient of variance determined. The averaged *.ost file was then converted into the IRIS format for mastercurve development. The files used to develop mastercurves in IRIS format were *.DDD and they included the test temperature, the frequency converted to radians/second, G', and G''. The standard deviation was calculated in the *.ost files to provide a confidence region for the data.

<u>Test Plan</u>

DSR Testing

The various parameters that can affect the characterization of materials using DSR, as discussed in the preceding sections, were used in the process of developing the characterization test plan. The sample preparation and conditioning included the following steps:

a)   Obtain silicone sealant materials from various manufacturers. A total of six silicone sealants from four manufacturers were tested.

b) Each sealant was poured into five molds which had a depth of 0.06 inches (1.5 mm). The length and width of the molds varied, but were typically 2.95 inches by 2.95 inches (75 mm by 75 mm).

c) Two of the molds were cured at laboratory conditions for 21 days before additional conditioning or testing.

d) Two of the molds were placed in a partially filled with water, loosely covered container. The container was placed in an oven set at 158°F (70°C) for 24, 48, or 72 hours.

e) Once the selected curing was completed, the samples were either tested, placed in the forced-draft oven for 7 or 14 days, or placed in the PAV for 24 or 72 hours.

f) After the selected curing and conditioning was completed, the samples were DSR tested.

The DSR testing procedures of the laboratory prepared silicone sealants consisted of the following:

a) A strain sweep was conducted using 0.98 inches (25 mm) diameter plates to determine the linear viscoelastic region of the silicone sample and to select test conditions that were within the constraints of the testing equipment.

b) One sample was placed in the DSR environmental chamber on the 0.98 inches (25 mm) diameter plates and adjusting the gap opening.

c) The temperature of the DSR environmental chamber was set to the desired test temperature. Once the chamber equilibrated at the test temperature, an additional five minute wait was allowed to ensure the sample had equilibrated at the test temperature.

d) The DSR was programmed to create sinusoidal deformation at frequencies of 0.02, 0.04, 0.06, 0.08, 0.1, 0.2, 0.4, 0.6, 0.8, 1.0, 2.0, 4.0, 6.0, 8.0, and 10.0 Hz. The sample was tested at each of these

frequencies, allowed a relaxation time and then the test sequence was repeated three additional times.

e) After testing had completed at the first test temperature, the environmental chamber was set at a temperature 18°F (10°C) higher, allowed to equilibrate, and the sample was tested at the 15 frequencies again. The process was continued until all test temperatures from -22 to 122°F (-30 to 50°C) in 18°F (10°C) increments was completed.

f) A second sample of the same material with the same conditioning was then tested using the same procedures as the first sample.

Data Analysis

Once the data had been collected, a multiphase effort was developed to analyze the results. The steps of the multiphase effort included:

a) The data files of the two similar samples were combined. The first measurement of each sample was rejected and the remaining measurements (six total) were averaged. The standard deviation and coefficient of variation of these measurements were also calculated.

b) The averaged data was examined to ensure that the measurement at each frequency had a range greater than one percent and a correction less than 0.2. Any measurement that did not meet this requirement was deleted from the data set.

c) The corrected data files were then converted into the IRIS 5 format and mastercurves at each of the test temperatures were developed using the automatic shift feature of the IRIS 5 software. The mastercurves were then evaluated to verify the automatic shifting had been accomplished in a logical manner. If large gaps were present in the mastercurves, the data were shifted manually.

d) Once the mastercurves were satisfactorily developed, G_∞ was estimated, subtracted from G', and the modified G' and original G'' data were converted to a discrete stress relaxation spectrum using the IRIS 5 software.

e) The estimated G_∞ and the g_i and λ_i from the discrete relaxation spectra were used as the viscoelastic input into the ABAQUS finite element code.

Poisson's Ratio Calculation

The Poisson's ratio (υ) for the joint sealant materials was determined using an image analyzer. Joint sealant samples were prepared in accordance with ASTM D412, Die C. The samples were allowed to cure for a minimum of 30 days before the "bow tie" specimens were cut from them. The specimens were then placed on a light stand and a picture was taken with a video camera. The picture was digitized on a workstation and measurements of the gage length and width were made using feature analysis software included with the image analysis system. The specimen was then stretched approximately 0.1 inches (2.54 mm) and another picture was taken and digitized. The measurements from the before and after picture were used to calculate the υ. A total of six samples were measured for each sealant material.

Model Verification

Perhaps the most important part of the silicone characterization investigation was the verification of the model that was developed through the characterization. To verify the ABAQUS model, two bond samples of sealant C were prepared using 7 inches (178 mm) long by 3 inches (76 mm) wide by 1 inch (25.4 mm) mortar blocks. Two mortar blocks were positioned such that there was a 0.5 inches (12.7 mm) opening between them. Spacers were positioned in the opening to allow a 0.5 inch (12.7 mm) wide by 0.5 inch (12.7 mm) thick by 7 inches (178 mm) long sealant bead was produced. The bead of sealant was allowed to cure for a minimum of 30 days before it was tested. After curing, the samples were placed in the Instron machine and extended at a rate of 0.125 inch (3.18 mm) per minute to an elongation

of 100 percent. The load required to extend the samples to 25, 50, 75, and 100 percent elongation was recorded. The test temperature for the tensile testing was 77°F (25°C). The load from these tests was converted to load per unit length and then compared to the ABAQUS modeling results.

CHAPTER 4 - SEALANT MATERIAL TESTING

All of the sealants were tested using the dynamic shear rheology (DSR) techniques provided in Chapter 3. As a result of that testing, several characteristics of the materials were determined with respect to temperature sensitivity and rate of loading effects. In this chapter, the results obtained for each of sealant and the statistical analysis of the data will be discussed

The DSR data can be presented in several formats but perhaps the two most beneficial methods are plots of shear storage modulus versus frequency and shear storage modulus versus temperature. The majority of stresses applied to a joint sealant will be the cyclic movement of the joint caused by temperature and/or moisture gradients in the slab. From this perspective, the shear storage modulus data would be better presented versus temperature instead of frequency. However, the frequency versus modulus mastercurves were used to develop the discrete stress relaxation spectra which provide the finite element modeling input. Only one frequency was selected for data presentation and discussion purposes; 1.0 Hz. A frequency of 1 Hz was selected to simplify the statistical analysis and because some research [53] has indicated that a dynamic loading of 1 Hz was approximately representative of the dynamic loading on an asphalt pavement caused by a vehicle traveling 55 mph (88.6 kph). There was no known correlation between this pavement loading and the loading that would be placed on the sealant in a concrete pavement but it provided a point of comparison between the various curing and aging conditions.

Typical Properties

The sealant labeling was selected arbitrarily with the exception that the non-sag type materials were grouped together and the self-leveling materials were grouped together. The trade names of the products were not mentioned in this report because the disclosure of those names would not enhance the results or conclusions of this research project. However, the following brief description of the products as provided in the manufacturer's product literature and material safety data sheet (MSDS) were provided to provide some insight into potential causes of test result differences.

Chemical Composition

Sealant A was a room temperature vulcanizing (RTV), oxime curing, low modulus, non-sag, polydimethylsiloxane based sealant. The sealant material also contained fillers, crosslinkers, and other chemicals which were considered to be a trade secret. The material produced a total of 4% methyl-ethylketoxime during vulcanization. Typical physical properties, as claimed by the manufacturer, of the uncured and cured material are provided in Table C.1 of Appendix C. This material had been installed on a limited number of projects on a trial basis.

Sealant B was a RTV, non-sag, polydimethylsiloxane based sealant. Other pigments and fillers may also be included in the sealant formulation. Typical physical properties as claimed by the manufacturer are provided in Table C.2 of Appendix C. This material had a history of satisfactory field performance.

Sealant C was a RTV, nog-sag, polydimethylsiloxane based, low modulus sealant. Other fillers and pigments may also be included in the sealant formulation. Typical physical properties as claimed by the manufacturer are provided in Table C.3 of Appendix C. This material had a history of satisfactory field performance.

Sealant D was a RTV, benzamide curing, non-sag, polydimethylsiloxane based sealant. The material also contained amorphous silicone dioxide, limestone, titanium, bis(N-methylbenzamido) methylethoxysilane, and a proprietary organo-titanate which was considered a trade secret. Other pigments and fillers may also be included in the sealant formulation. Typical physical properties as claimed by the manufacturer are provided in Table C.4 of Appendix C. This material had a limited history of satisfactory field performance.

Sealant E was a RTV, dimethylpolysiloxane based, self-leveling, low modulus sealant. The sealant also contained alkyloximino silane and toluene in addition to fillers and pigments. Typical physical properties as claimed by the manufacturer are provided in Table C.5 of Appendix C. The sealant had a history of satisfactory field performance.

Sealant F was a RTV, polydimethylsiloxane based, self-leveling, ultra-low modulus sealant which evolved n-methylacetamide when exposed to water or humid air. The sealant also contained methylvinyl bis(n-methlacetamido)silane, and calcium carbonate which had been treated with steric acid. Additionally, the sealant may contain other fillers, pigments, and crosslinkers. Typical physical properties as claimed by the manufacturer are provided in Table C.6 of Appendix C. This material had a history of satisfactory field performance.

The brief summary of the sealant materials indicates that they were all polydimethylsiloxane based, moisture curing, low to ultra-low modulus materials, and they have all exhibited satisfactory field performance on one or more projects. It was also evident that they are a mixture of components which included the base material, fillers and pigments.

Physical Properties

The typical physical properties for the materials presented in Appendix C indicated some differences in the cured materials. The elongation of unaged samples at failure ranged 700 percent to approximately 1600 percent and the elastic modulus values at 150 percent elongation ranged from 10 to 45 psi (69 to 310 KPa). There were also some small differences in the Shore A hardness results of the non-sag materials (sealants A through D). However, the differences in Shore 00 hardness results of the self-leveling materials (sealants E and F) were somewhat larger. A summary of the physical properties is provided in Table 4.1. These typical physical properties as provided by the manufacturers indicated that some differences should be noted in the DSR analysis. The data provided by the manufacturers did not include aging data. Instead the manufacturers state that the sealants are resistant to aging and weathering.

DSR Storage Shear Modulus Versus Temperature Results

One of the more important considerations of joint sealant field performance was the effect of temperature on the material properties of the sealant. Therefore,

the first data analyzed were the storage shear modulus ($G^/$) versus temperature data. The 1 Hz, $G^/$ data for all of the sealant materials are provided in Appendix D.

Table 4.1. Sealant Summarized Physical Properties.

Sealant	Type	Elongation to Failure (Percent)	Modulus at 150% Elongation (psi)	Shore Hardness
A	Non-Sag	800	Not Provided	16[1]
B	Non-Sag	958	38.9	11[1]
C	Non-Sag	1200	45.0	15[1]
D	Non-Sag	700	43.0	15[1]
E	Self-Leveling	884	22.1	64[2]
F	Self-Leveling	1600	10.0	40[2]

[1] Shore A test results.
[2] Shore 00 test results.

Sealant A DSR Results

The summarized results for sealant A are provided in Tables D.1 through D.4 and generalized trends for sealant A are provided in Tables 4.1 through 4.3. The trends presented in Table 4.2 indicated that the $G^/$ of the sealant material, regardless of curing time or aging condition, initially remained constant or exhibited a stiffening to some temperature between -4°F (-20°C) and 50°F (10°C). Above that temperature, $G^/$ decreased with increasing temperatures. The trend exhibited in these tests was not expected. The expected trend was either no change in stiffness or a decrease in stiffness with increasing test temperatures because for most viscoelastic materials, increases in temperature would result in a softening of the material (i.e., a decrease in $G^/$). The significance of the trends provided in Table 4.2 was somewhat questionable. The variability in the $G^/$ values suggested that the

stiffness of the material may not change with changes in test temperatures. This could be illustrated by averaging the G' values at all of the temperatures within a specific conditioning and then calculating the standard deviation. For example, the average G' for the unaged 24 hour accelerated cured sealant A material would be 7.37 psi (50.8 KPa) with a standard deviation of 1.45 psi (10.0 KPa). Establishing a 95 percent confidence region around 7.37 psi (50.8 KPa) would include all of the average responses listed for each temperature within that condition. Based upon this brief analysis, it is evident that an increase in stiffness of the silicone sealant A with an increase in temperature may not be representative of the material.

Table 4.3 provides the generalized trends of sealant A with respect to the length of time the material was aged. It was expected that as the sealant material was aged, the G' would increase indicating that the material was becoming stiffer. This trend was exhibited in the 24 hour accelerated cured material in which the G' increased when comparing the unaged material to the 7 day oven aged material and comparing the 7 day oven aged material to the 14 day oven aged material. The expected trend did not hold true for the other curing conditions tested. The 48 hour accelerated cured, 14 day oven aged material exhibited a decrease in G' when compared to both the unaged and 7 day oven aged material. However, the 48 hour accelerated cured, 7 day oven aged material exhibited an increase in G' as compared to the unaged material. The 21 day laboratory cured material exhibited the same general trend as the 48 hour accelerated cured material, but the 72 hour accelerated cured material exhibited an almost opposite trend. The 72 hour accelerated material appeared to soften when comparing the unaged material to the 7 day oven aged material and stiffened when comparing the unaged material to the 14 day oven aged material and the 7 day oven aged material to the 14 day oven aged material. The exception to this trend was the 48 and 72 hour accelerated cured materials which exhibited very similar modulus versus temperature responses. The 48 and 72 hour accelerated cured materials and the 21 day laboratory cured material generally exhibited a decrease in G' as the temperature increased.

Table 4.2. Generalized Trends of G' for Sealant A With Temperature.

Condition	Temperature ↗	
24 Hour Accelerated Cure, Unaged	↗to 0°C	↘ above 0°C
24 Hour Accelerated Cure, 7 Day Oven Aged	↗ to -10°C	↘ above -10°C
24 Hour Accelerated Cure, 14 day Oven Aged	→ to 10°C	↘ above 10°C
48 Hour Accelerated Cure, Unaged	↗ to -20°C	↘ above -20°C
48 Hour Accelerated Cure, 7 Day Oven Aged	↗ to 0°C	↘ above 0°C
48 Hour Accelerated Cure, 14 Day Oven Aged	↗ to -10°C	↘ above -10°C
72 Hour Accelerated Cure, Unaged	↗ to 0°C	↘ above 0°C
72 Hour Accelerated Cure, 7 Day Oven Aged	↗ to 0°C	↘ above 0°C
72 Hour Accelerated Cure, 14 Day Oven Aged	↗ to -10°C	↘ above -10°C
21 Day Laboratory, Unaged	↗ to -10°C	↘ above -10°C
21 Day Laboratory Cure, 7 Day Oven Aged	↗ to 0°C	↘ above 0°C
21 Day Laboratory Cure, 14 Day Oven Aged	↗ to -20°C	↘ above -20°C

Table D.2 provides the summarized data for the 7 day oven aged samples. The 7 day oven aged sample trends presented in Table 4.4 indicated a stiffening or an increase in G' for each of the curing conditions as compared to the unaged samples with the exception of the 72 hour accelerated cured samples. The average value G' of the 72 hour accelerated cured samples remained basically unchanged. However, the standard deviation for the 72 hour accelerated cured samples that had been oven aged for 7 days was much larger than any of the other 7 day oven aged

samples or the unaged samples. The coefficient of variation for the 72 hour accelerated cured samples were approximately 50 to 55 percent while the coefficient of variation for the unaged samples and other 7 day oven aged samples were generally less than 20 percent. The increase in G' due to increased curing time as noticed in the unaged samples was not evident in the 7 day oven aged samples.

Table 4.3. Generalized Trends of G' for Sealant A Aging.

Amount of Aging	24 Hour Accelerated Cure	48 Hour Accelerated Cure	72 Hour Accelerated Cure	21 Day Laboratory Cure
Unaged to 7 Day Oven Aged	↗	↗	↘	↗
Unaged to 14 Day Oven Aged	↗	↘	↗	↘
7 Day Oven Aged to 14 Day Oven Aged	↗	↘	↗	↘

The generalized trend for the 7 day oven aged samples was a decrease in the G' with an increase in temperature for temperatures above 32°F (0°C). In general, the G' increased with increasing temperatures at temperatures below 32°F (0°C). It was expected that the data would exhibit a decrease in the G' with an increase in temperature for all of the test temperatures. However, averaging the data from all test temperatures within a given curing condition and establishing a 95 percent confidence region around that average indicated that a single G' value could be selected for each curing condition. The fact that the 95 percent confidence region incorporated all of the data for each temperature within a specific curing condition could infer that the positive relationship of G' to temperature may not be representative of the material properties.

The data from the 14 day oven aged samples, shown in Table D.3, at each of the curing conditions exhibited the same general trend (provided in Table 4.4) as the unaged and 7 day oven aged where the G' increased with increases in the test temperature to approximately 32°F (0°C). At temperatures above 32°F (0°C), the G' decreased with increasing temperature. The 14 day oven aged samples did not exhibit an additional increase in G' with aging and in fact resembled more closely to the unaged sample results. It was expected that the G' values for the 14 day oven aged results would be similar to or higher than the 7 day oven aged results.

Table 4.4. Generalized Trends of G' for Sealant A With Length of Cure.

Length of Cure	Unaged Samples	7 Day Oven Aged Samples	14 Day Oven Aged Samples
24 Hours Accelerated Cure to 48 Hours Accelerated Cure	↗	↘ to -10°C ↗ above -10°C	↘
24 Hours Accelerated Cure to 72 Hours Accelerated Cure	↗	↘	↘
24 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗	↘
48 Hours Accelerated Cure to 72 Hours Accelerated Cure	↘	↘	↗
48 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↘ to -10°C ↗ above -10°C	↗
72 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗	↘

The coefficient of variation of the various control conditions of sealant A ranged from approximately 1 percent to 57 percent. However, approximately 83 percent of the control conditions had a coefficient of variation less than 30 percent, approximately 73 percent of the control conditions had a coefficient of variation of

less than 20 percent, and approximately 30 percent of the control conditions had a coefficient of variation of less than 10 percent. One of the control conditions was length of cure, it was expected that the coefficient of variation would decrease as the length of cure increased. This trend was expected because the curing mechanism of the sealant progressed from the exposed surface downward. Any variations in sample thickness could create samples in which the degree of cure varied, and, therefore, the viscous and elastic responses of the material would vary. As the length of cure increased, the samples should obtain full cure and the viscous and elastic responses would become more uniform. The test data for sealant A did not follow this trend. Instead, the coefficient of variation appeared to be random.

Sealant B DSR Test Results

The summarized data for sealant B are provided in Tables D.5 through D.7 and the generalized trends for sealant B are provided in Tables 4.5 through 4.7. The majority of the test results indicated that G' for each of the control conditions exhibited the same generalized trend versus temperature as sealant A. The generalized trend exhibited was either no change or an increase in G' to some specific temperature between -4 and 50°F (-20 and 10°C), and then decreased as the test temperature increased. The 24 hour accelerated cured, unaged and the 72 hour accelerated cured, 7 day oven aged samples exhibited a decrease in G' over the entire temperature range and the 72 hour accelerated cured, 14 day oven aged sample remained virtually constant over the entire temperature range. These three samples exhibited the generalized trend that was expected for all the materials. Data was not available for all of the test temperatures for all of the control conditions. Some of the raw data files were damaged during the software manipulation and some samples were damaged during preparation before testing.

The generalized trends of sealant B with respect to aging are provided in Table 4.6. The 24 hour accelerated cure exhibited the expected generalized trend which was an increase in G' as the length of aging increased. The 48 hour accelerated cure also exhibited an increase in G' with an increase in the length of

Table 4.5. Generalized Trends of G′ for Sealant B With Temperature.

Condition	Temperature ↗
24 Hour Accelerated Cure, Unaged	↘ over testing range
24 Hour Accelerated Cure, 7 Day Oven Aged	↗ to -10°C ↘ above -10°C
24 Hour Accelerated Cure, 14 day Oven Aged	↗ to -20°C ↘ above 20°C
48 Hour Accelerated Cure, Unaged	→ to 10°C ↘ above 10°C
48 Hour Accelerated Cure, 7 Day Oven Aged	No data ↘ above 10°C below 10°C
48 Hour Accelerated Cure, 14 Day Oven Aged	No data
72 Hour Accelerated Cure, Unaged	↗ to 0°C ↘ above 0°C
72 Hour Accelerated Cure, 7 Day Oven Aged	↘ No data for -10, 30, 40, or 50°C
72 Hour Accelerated Cure, 14 Day Oven Aged	→ over testing range
21 Day Laboratory, Unaged	→ to 10°C ↘ above 10°C
21 Day Laboratory Cure, 7 Day Oven Aged	↘ No data above 30°C
21 Day Laboratory Cure, 14 Day Oven Aged	→ to 10°C ↘ above 10°C

aging for the data that was available. The 48 hour accelerated cured, 14 day oven aged samples were damaged during preparation and therefore comparisons between the unaged and 7 day oven aged versus the 14 day oven aged samples could not be conducted. The 72 hour accelerated cured material exhibited almost no change in stiffness with aging. Some fluctuation did occur within specific temperatures but overall, the stiffness remained relatively constant. The 21 day laboratory cured

material exhibited an increase in stiffness upon aging when comparing the unaged samples to both the 7 day and 14 day oven aged samples. Comparisons between the 21 day laboratory cured 7 day and 14 day oven aged samples indicated that below 68°F (20°C), G' decreased with age but above 68°F (20°C), G' increased. The expected trend for G' versus aging was either no change or an increase in G' as the length of aging was increased.

Table 4.6. Generalized Trends of G' for Sealant B Aging.

Amount of Aging	24 Hour Accelerate d Cure	48 Hour Accelerate d Cure	72 Hour Accelerate d Cure	21 Day Laborator y Cure
Unaged to 7 Day Oven Aged	↗	↗	→	↗
Unaged to 14 Day Oven Aged	↗	No data available	→	↗
7 Day Oven Aged to 14 Day Oven Aged	↗	No data available	→	↘ to 20°C ↗ above 20°C

The generalized trend for sealant B comparing G' to length of cure is provided in Table 4.7. The data indicated that the trend was basically the same regardless of the amount aging to which the sample was exposed. All of the sealant B, 48 hour accelerated cure samples exhibited a higher G' than the 24 hour accelerated cured materials. This same trend was exhibited when comparing the 21 day laboratory cured samples to the 24, 48, and 72 hour accelerated cured materials. Comparing the 72 hour accelerated cured material to the 24 and 48 hour materials exhibited a different trend than expected. The comparison between the 72 and 24 hour accelerated cured, unaged material exhibited no change but the 7 and 14 day oven aged samples exhibited a decrease in G' with increased curing time. In

addition, when comparing the 72 and 48 hour accelerated cured samples, a decrease in G' was exhibited with an increase in curing time. The expected trend was an increase in G' with an increase in the length of curing up to some maximum G' value above which additional curing would not increase G'.

Table 4.7. Generalized Trends of G' for Sealant B With Length of Cure.

Length of Cure	Unaged Samples	7 Day Oven Aged Samples	14 Day Oven Aged Samples
24 Hours Accelerated Cure to 48 Hours Accelerated Cure	↗	↗ for available data	No data available
24 Hours Accelerated Cure to 72 Hours Accelerated Cure	→	↘ for available data	↘
24 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗ for available data	↗
48 Hours Accelerated Cure to 72 Hours Accelerated Cure	↘	↘ for available data	No data available
48 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗ for available data	No data available
72 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗ for available data	↗

The coefficient of variation for sealant B was less than that calculated for sealant A, but in general, it would be possible to represent G' for most of the control

conditions as a single value. The coefficient of variation ranged from approximately 1 to 40 percent. Therefore, the validity of some of the generalized trends may not be significant. Approximately 95 percent of the coefficients of variation were less than 30 percent, approximately 90 percent of the coefficients of variation were less than 20 percent, and approximately 64 percent of the coefficients of variation were less than 10 percent. No trend was evident in the coefficients of variation, i.e., they did not decrease with increased length of cure or age.

Sealant C DSR Test Results

The summarized data for sealant C are provided in Tables D.8 through D.10 and the generalized trends are provided in Tables 4.8 through 4.10. The G' data for sealant C within a specific control condition versus temperature was more variable than for sealant A or B. For example the 24 hour accelerated cured data cycled between consecutive temperatures; G' was 9.1 psi (63 KPa) at -22°F (-30°C), 8.7 psi (60 KPa) at -4°F (-20°C), and 9.0 psi (62 KPa) at 14°F (-10°C), etc. Even with the increased variability, the generalized trend was similar to sealants A and B. The data exhibited either no change or an increase in G' to some specific temperature and then exhibited a decrease in G' as the test temperature continued to increase. The exceptions to this trend were the 24 hour accelerated cured, 7 day oven aged; the 48 hour accelerated cured, unaged; and the 21 day laboratory cured, 14 day oven aged materials. The 24 hour accelerated cured, 7 day oven aged material exhibited no change in G' from -22 to -4°F (-30 to -20°C), a decrease in G' to 14°F (-10°C), and then an increase in G' at test temperatures above 14°F (-10°C). The 48 hour accelerated cured, unaged material exhibited an increase in G' over the entire test temperature range and the 21 day laboratory cured, 14 day oven aged material exhibited an increase in G' from -22 to -4°F (-30 to -20°C), remained constant from approximately -4 to 50°F (-20 to 10°C), and then decreased at test temperatures above 50°F (10°C). The expected trend for G' versus temperature was that as the test temperature was increased, G' would decrease.

Table 4.8. Generalized Trends of G′ for Sealant C With Temperature.

Condition	Temperature ↗
24 Hour Accelerated Cure, Unaged	→ over testing range
24 Hour Accelerated Cure, 7 Day Oven Aged	→ to -20°C ↘ to -10°C ↗ above -10°C
24 Hour Accelerated Cure, 14 day Oven Aged	↗ to 30°C ↘ above 30°C
48 Hour Accelerated Cure, Unaged	↗ over testing range
48 Hour Accelerated Cure, 7 Day Oven Aged	↗ to 20°C · ↘ above 20°C
48 Hour Accelerated Cure, 14 Day Oven Aged	↗ to 0°C ↘ above 0°C
72 Hour Accelerated Cure, Unaged	↘ over testing range
72 Hour Accelerated Cure, 7 Day Oven Aged	→ to 20°C ↘ above 20°C No data for -30 or -20°C
72 Hour Accelerated Cure, 14 Day Oven Aged	↗ to 0°C ↘ above 0°C
21 Day Laboratory, Unaged	↘ over testing range
21 Day Laboratory Cure, 7 Day Oven Aged	↗ to 0°C ↘ above 0°C
21 Day Laboratory Cure, 14 Day Oven Aged	↗to -20°C → to 10°C ↘ above 10°C

The generalized trend of G′ versus aging is presented in Table 4.9. The data indicated that as the material was aged for 7 days in the oven, G′ generally decreased regardless of the curing condition. A decrease in G′ was also exhibited in the initial data when comparing the 24 hour accelerated cured unaged material to the 14 day oven aged material, the initial data of the 24 hour accelerated cured 7 day oven aged to the 14 day oven aged material, and over the complete test temperature range of the 48 hour accelerated cure unaged material as compared to

the 14 day oven aged material. The other remaining control conditions exhibited the expected trend of either no change or an increase in G' as the length of aging was increased.

Table 4.9. Generalized Trends of G' for Sealant C Aging.

Amount of Aging	24 Hour Accelerated Cure	48 Hour Accelerated Cure	72 Hour Accelerated Cure	21 Day Laboratory Cure
Unaged to 7 Day Oven Aged	↘	↘	↘	↘
Unaged to 14 Day Oven Aged	↘ to 10°C ↗ above 10°C	↘	↗	→
7 Day Oven Aged to 14 Day Oven Aged	↘ to -20°C ↗ above -20°C	→to -10°C ↗ above -10°C	↗	→to 20°C ↗ above 20°C

The trends associated with changes in G' as it related to the length of sample curing is provided in Table 4.10. The data exhibited the expected trend for the majority of the data. The expected trend was an increase in stiffness with increasing cure times. The exceptions to this trend were the 7 day oven aged 24 hour accelerated cured at test temperatures above 50°F (10°C) as compared to the 48 hour accelerated cured material and both the unaged and 7 day oven aged 24 hour accelerated cured materials as compared to the 21 day laboratory cured materials.

Sealant C exhibited an increase in variability as compared to sealants A and B. The coefficients of variability for sealant C ranged from approximately 3 percent to 79 percent. Approximately 69 percent of the coefficients of variation were less than 30 percent, approximately 47 percent were less than 20 percent, and approximately 12 percent were less than 10 percent. The magnitude of the

coefficients of variability indicated that the generalized trends exhibited by sealant C may not be significant.

Table 4.10. Generalized Trends of G' for Sealant C With Length of Cure.

Length of Cure	Unaged Samples	7 Day Oven Aged Samples	14 Day Oven Aged Samples
24 Hours Accelerated Cure to 48 Hours Accelerated Cure	↗	↗	↗
24 Hours Accelerated Cure to 72 Hours Accelerated Cure	↗	↗ to 10°C ↘ above 10°C	↗
24 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗	↗
48 Hours Accelerated Cure to 72 Hours Accelerated Cure	↘	↘	↗
48 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗	↗
72 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↗	↗

The coefficients of variation were expected to decrease as the technician became more familiar with the test equipment and the sample preparation techniques. If the coefficient of variation was limited to the learning curve of the technician, then sealant C should have similar or smaller coefficients of variation as compared to sealant B. One potential cause of the increased variability was the fact that sealant C appeared to have a higher viscosity than sealants A and B. Therefore, additional care would be required during the preparation of sealant C samples. Additional tests were conducted using sealant C in an attempt to investigate the variability. The results from this testing are presented in Table D.18. These test

results indicated that a larger number of samples did reduce variability in the sealant C test results. This testing indicated that more than two samples would be required to adequately characterize the sealant materials.

Sealant D DSR Test Results

The summarized data for sealant D are presented in Tables D.11 through D.13 and the generalized trends are provided in Tables 4.11 through 4.13. The generalized G' versus test temperature trends for sealant D were similar to those exhibited in sealants A and B. All test conditions exhibited an increase in G' initially to some specific temperature (-4 to 68°F (-20 to 20°C)) and then either no change or a decrease in G' as the test temperatures continued to increase. The variability in the sealant D test results was less than the variability exhibited in sealant C, but the establishment of a 95 percent confidence region around the results indicated that a single G' value could be used to characterize the materials at each condition.

Table 4.12 provides the generalized trends of G' versus length of aging. The aging trends were similar to those that would be expected. As the length or amount of aging was increased, G' increased. The exceptions to this trend were the comparisons between the 48 hour accelerated cured material at 7 and 14 day oven aging and the 21 day laboratory cured samples comparing the unaged material to the 14 day oven aged material and comparing the 7 day oven aged material to the 14 day oven aged material.

The trends associated with the length of curing were somewhat reversed from what would be expected. Sealant D appeared to become softer as the cure time was increased. The exceptions to this trend were the 48 to 72 hour accelerated cured unaged samples, the 24 to 48 and the 24 to 72 hour accelerated cured 7 day oven aged samples, and the 24 to 48, the 24 to 72, and the 48 to 72 hour accelerated cured 14 day oven aged samples.

Table 4.11. Generalized Trends of G$^{/}$ for Sealant D With Temperature.

Condition	Temperature ↗
24 Hour Accelerated Cure, Unaged	↗ to 10°C ↘ above 10°C
24 Hour Accelerated Cure, 7 Day Oven Aged	↗ to 0°C ↘ above 0°C
24 Hour Accelerated Cure, 14 day Oven Aged	↗ to -10°C → to 20°C ↘ above 30°C
48 Hour Accelerated Cure, Unaged	↗ to -20°C ↘ above -20°C
48 Hour Accelerated Cure, 7 Day Oven Aged	↗ to -10°C ↘ above -10°C
48 Hour Accelerated Cure, 14 Day Oven Aged	↗ to 10°C ↘ above 10°C
72 Hour Accelerated Cure, Unaged	↗ to 10°C ↘ above 10°C
72 Hour Accelerated Cure, 7 Day Oven Aged	↗ to -10°C ↘ above -10°C
72 Hour Accelerated Cure, 14 Day Oven Aged	↗ to 0°C ↘ to 10°C → above 10°C
21 Day Laboratory, Unaged	↗ to 20°C → above 20°C
21 Day Laboratory Cure, 7 Day Oven Aged	↗ to 20°C → above 20°C
21 Day Laboratory Cure, 14 Day Oven Aged	↗ to 0°C → above 0°C

The coefficients of variation for sealant D ranged from approximately 1 percent to approximately 50 percent. The variability was similar to that exhibited in the test results of sealant A. Approximately 91 percent of the coefficients of variability were less than 30 percent, approximately 77 percent were less than 20 percent, and approximately 22 percent of the coefficients of variation were less than 10 percent.

Table 4.12. Generalized Trends of G' for Sealant D Aging.

Amount of Aging	24 Hour Accelerated Cure	48 Hour Accelerated Cure	72 Hour Accelerated Cure	21 Day Laboratory Cure
Unaged to 7 Day Oven Aged	↗	↗	↗	↗
Unaged to 14 Day Oven Aged	↗	↗	↗	↘
7 Day Oven Aged to 14 Day Oven Aged	↗	↘	↗	↘

Table 4.13. Generalized Trends of G' for Sealant D With Length of Cure.

Length of Cure	Unaged Samples	7 Day Oven Aged Samples	14 Day Oven Aged Samples
24 Hours Accelerated Cure to 48 Hours Accelerated Cure	↘	↗	↗
24 Hours Accelerated Cure to 72 Hours Accelerated Cure	↘	↗	↗
24 Hours Accelerated Cure to 21 Days Laboratory Cure	↘	↘	↘
48 Hours Accelerated Cure to 72 Hours Accelerated Cure	↗	↘	↗
48 Hours Accelerated Cure to 21 Days Laboratory Cure	↘	↘	↘
72 Hours Accelerated Cure to 21 Days Laboratory Cure	↘	↘	↘

Sealant E DSR Test Results

The summarized results for sealant E are provided in Table D.14 and the generalized trends are provided in Tables 4.14 through 4.15. The samples that were exposed to the accelerated curing conditions did not cure, and, therefore, were not tested. A second sample of sealant E was obtained from the manufacturer and specimens were prepared for accelerated curing. This second set of specimens also did not cure. The fact that neither set of samples cured implied that the accelerated curing technique could not be used as a specification technique because it would eliminate the use of at least one of the currently available sealants. If the sealant had a history of poor performance, the accelerated curing technique could potentially be used to delineate between satisfactory and non-satisfactory materials. However, sealant E had a history of performing satisfactorily in the field and as such the accelerated curing technique would not be recommended for specification use.

The exhibited trend of G' versus temperature for each of the conditions was similar to those exhibited by the previous sealants. The material exhibited an initial stiffening up to -4 or 14°F (-20 or -10°C) and then remained constant as the test temperature was increased. Establishing a 95 percent confidence region for each of the test results indicated that a single value could be determined to characterize the sealant.

Table 4.14. Generalized Trends of G' for Sealant E With Temperature.

Condition	Temperature ↗
21 Day Laboratory, Unaged	↗ to 10°C → above 10°C
21 Day Laboratory Cure, 7 Day Oven Aged	↗ to -20°C → above -20°C
21 Day Laboratory Cure, 14 Day Oven Aged	↗ to -10°C → above -10°C

The comparisons between the unaged and 7 day oven aged and between the unaged and 14 day oven aged samples indicated an increase or no change in G' with an increase in the length of aging. Comparisons between the 7 day oven aged and 14 day oven aged samples; however, indicated a decrease in G' with an increase in aging. The expected trend would be an increase in G' with increased length of aging.

Table 4.15. Generalized Trends of G' for Sealant E Aging.

Amount of Aging	21 Day Laboratory Cure
Unaged to 7 Day Oven Aged	↗
Unaged to 14 Day Oven Aged	→
7 Day Oven Aged to 14 Day Oven Aged	↘

The coefficients of variation for sealant E ranged from approximately 9 percent to approximately 38 percent. Approximately 74 percent of the coefficients of variation were less than 30 percent, approximately 37 percent were less than 20 percent, and approximately 7 percent of the coefficients of variation were less than 10 percent. These results indicated that the variability of sealant E was similar to those exhibited by sealant C. Additionally, the coefficients of variation indicated that the variability was not related simply to the technician's learning curve because this was one of the last sealants tested. If the variability was due to the learning curve, it would have been less than those exhibited for sealants A and B.

Sealant F DSR Test Results

The summarized test results for sealant F are provided in Tables D.15 through D.17 and the generalized trends are provided in Tables 4.16 through 4.18. The G' trends versus temperature for sealant F shown in Table 4.16 were similar to the trends exhibited by the previous sealants, i.e., increased to some specific temperature

and then decreased over the remaining test temperatures. There were four conditions; the unaged, 24 and 48 hour accelerated cured materials and the 7 day oven aged, 24 and 48 hour accelerated cured materials, which exhibited a decrease in G' over the majority of the test temperatures. The expected trend for this sealant was a decrease or no change in G' with increasing test temperatures.

The trends associated with aging sealant F are provided in Table 4.17. The expected trend of G' with increased length of aging was no change or an increase in G'. This expected trend was exhibited in over half of the testing condition comparisons. The remaining test condition comparisons exhibited an increase in G' with an increase in the length of aging.

Table 4.18 provides the generalized trends for G' as compared to the length of cure for the sealant samples. The expected trend for G' versus length of cure was that as the length of cure increased, G' should increase. Five of the testing conditions exhibited this expected trend, four testing conditions exhibited no change in G', and seven of the testing conditions, all of the 7 day oven aged samples and the 14 day oven aged, 48 to 72 hour accelerated cured comparison, exhibited a decrease in G' with increases in length of aging.

The coefficients of variation for sealant F ranged from approximately 1 percent to approximately 71 percent. Approximately 78 percent of the coefficients of variation were less than 30 percent, approximately 62 percent were less than 20 percent, and approximately 44 percent were less than 10 percent. The amount of variation indicated that the generalized trends may not be representative of the physical properties of the material evaluated. The variation also indicated that the assumptions made by Tons [39] and Wang [47] concerning the insensitivity of silicone materials to aging and weathering could be sufficient for material modeling. Finally, the variation indicated that a larger sample population must be tested to assist in determining statistical significance of changes in G'.

Table 4.16. Generalized Trends of G' for Sealant F With Temperature.

Condition	Temperature ↗
24 Hour Accelerated Cure, Unaged	↘ over testing range
24 Hour Accelerated Cure, 7 Day Oven Aged	↘ over testing range
24 Hour Accelerated Cure, 14 day Oven Aged	↗ to -20°C ↘ to 20°C ↗ above 20°C
48 Hour Accelerated Cure, Unaged	↘ over majority of testing range
48 Hour Accelerated Cure, 7 Day Oven Aged	↘ over majority of testing range
48 Hour Accelerated Cure, 14 Day Oven Aged	↗ to -20°C ↘ above -20°C
72 Hour Accelerated Cure, Unaged	↗ to -20°C ↘ above -20°C
72 Hour Accelerated Cure, 7 Day Oven Aged	↗ to -20°C ↘ above -20°C
72 Hour Accelerated Cure, 14 Day Oven Aged	↗ to 10°C ↘ above 10°C
21 Day Laboratory, Unaged	↗ to 20°C ↘ above 20°C
21 Day Laboratory Cure, 7 Day Oven Aged	↗ to 10°C ↘ above 0°C
21 Day Laboratory Cure, 14 Day Oven Aged	↗ to -20°C → to 10°C ↘ above 10°C

Table 4.17. Generalized Trends of G' for Sealant F Aging.

Amount of Aging	24 Hour Accelerated Cure	48 Hour Accelerated Cure	72 Hour Accelerated Cure	21 Day Laboratory Cure
Unaged to 7 Day Oven Aged	↗	→	↘	↘
Unaged to 14 Day Oven Aged	↘	→	↗ above -10°C	→
7 Day Oven Aged to 14 Day Oven Aged	↘	→	↗	↘

Table 4.18. Generalized Trends of G' for Sealant F With Length of Cure.

Length of Cure	Unaged Samples	7 Day Oven Aged Samples	14 Day Oven Aged Samples
24 Hours Accelerated Cure to 48 Hours Accelerated Cure	→	↘	↗
24 Hours Accelerated Cure to 72 Hours Accelerated Cure	→	↘	→
24 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↘	↗
48 Hours Accelerated Cure to 72 Hours Accelerated Cure	→	↘	↘
48 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↘	↗
72 Hours Accelerated Cure to 21 Days Laboratory Cure	↗	↘	↗

Statistical Analysis of DSR Data

The shear storage modulus values determined for each sealant at each test temperature and condition exhibited widely varying coefficients of variation. As a result of the variability, the 95 percent confidence regions established around those results indicated that the differences between the measured shear storage modulus values were potentially not statistically significant. To determine if the differences were statistically significant, a more rigorous statistical analysis was required.

The statistical software package SigmaStat ™ was used to analyze the statistical significance of differences between the modulus data of the various sealant materials collected at the various conditions and test temperatures. Additional information on the capabilities of SigmaStat ™ can be obtained from Jandel Scientific Software [54].

Two areas were investigated using statistical analysis techniques. The first area of investigation was to determine if differences between the modulus values obtained from two or more samples at the same curing and aging conditions were statistically significant. Also during this phase of the investigation, temperature versus storage modulus trends for the samples were investigated to determine if consistency could be detected between the samples, i.e., if the storage modulus of the samples exhibited an increase, decrease, or remained unchanged versus temperature. The second area of the statistical analysis was to determine if differences in the average modulus values collected at the various test temperatures as well as between curing and aging conditions at the same test temperature were statistically significant. The data collected at a test frequency of 1 Hz was imported into SigmaStat ™ and either a one way repeated measures analysis of variance (ANOVA) or a one way ANOVA was selected to compare the data. The one way repeated measures ANOVA and the one way ANOVA were selected because only one factor (the test temperature, the curing condition, or the length of aging) was being varied between the samples that were being compared. The repeated measures ANOVA was used to analyze the individual samples because three measurements were taken on each sample. In the majority of cases, the test for normality failed using a P value of 0.05.

The reason the data failed the normality test was because the data generally had a bimodal distribution. The Student-Newman-Keuls (SNK) method for pairwise multiple comparisons was used after the one way ANOVA to identify those differences which were significant.

Sealant A Statistical Analysis

The statistical analysis of the sealant A samples which were exposed to the same curing and aging conditions and tested at the same temperature indicated that the differences between many of samples were statistically significant. Examining the graphical representation of the individual specimen data did not delineate any specific trends. For example, all of the differences between samples of the 24 hour accelerated cured material were statistically significant except for the first and second sample tested at 32°F (0°C). These two samples were taken from the same sheet of cured sealant material. However, the few differences exhibited in the individual samples at each test temperature of the 48 hour accelerated that were not statistically different were not necessarily taken from the same cured sheet of material. Also the graphical representation of the individual samples did not exhibit the same G' versus temperature trends. Some of the samples exhibited an increase in G' with temperature while other exhibited a decrease or no change with temperature. Additionally, some samples taken from the same sheet of cured material crossed with one sample (sample 1) having a lower modulus than the other sample (sample 2) at low temperatures but a higher modulus than the other sample at higher temperatures.

The fact that the differences were determined to be statistically different was expected. The data indicated that the three measurements for the majority of individual samples were almost identical yielding standard deviations of either zero or a very small number as compared to the storage modulus. The differences exhibited in the samples of the remainder of the various testing conditions were generally determined to be statistically significant.

The statistical analysis of the temperature data within a specific curing and aging condition provided inconsistent results. For this portion of the statistical analysis, all of the data obtained at a specific temperature and test condition were averaged. The analysis of the 24 hour accelerated cured, unaged, sealant A material indicated that the 1 Hz modulus values for the test temperatures ranging from -22°F to 14°F (-30°C to -10°C) could be combined (i.e., the differences were not statistically significant). The differences between the 32°F and 14°F (0°C and -10°C) data were not statistically significant and the differences between the 50°F through 122°F (10°C through 50°C) were not statistically significant. Combining the data whose differences were not statistically different indicated a slight change in the trend exhibited by the shear storage modulus with respect to temperature. Before combining the data, the exhibited trend was an increase in G' with an increase in temperature to 32°F (0°C) and then a decrease in G' as the temperature continued to increase. After the data combination, the exhibited trend was an increase in G' with increasing temperatures to 50°F (10°C) and then no change in G' as the test temperature continued to increase.

The combining of the data whose differences were not statistically significant appeared to be a logical step when considering the 24 hour accelerated cured, sealant A material. The reason behind the logic was that the data that were combined were those that had sequential test temperatures, i.e., the -22°F (-30°C) data was combined with the -4°F (-20°C) and 14°F (-10°C) data, the 32°F (0°C) data was combined with the 14°F (-10°C) data, and the 50°F (10°C) data was combined with the 68, 86, 104, and 122°F (20, 30, 40, and 50°C). The data combinations delineated through the statistical analysis of the 48 hour accelerated cured, unaged, sealant A material were not so logical. For example, the statistical analysis indicated that the data for the 48 hour accelerated cured, unaged sealant A material did not exhibit a statistically significant difference between the -4°F (-20°C) data and the 32, 50, 68, 86, 104, and 122°F (0, 10, 20, 30, 40, and 50°C) data. However, the difference between the -4°F (-20°C) and the -22 and 14°F (-30 and -10°C) data were statistically

significant. The statistical analysis of the 48 hour accelerated cured, unaged sealant A material indicated that G' at -4°F (-20°C) and 68°F (20°C) were the same but G' was greater at test temperatures between -4 and 68°F (-20 and 20°C). The trend exhibited by the statistically combined 48 hour accelerated cured, unaged sealant A material was the same as that exhibited by the original data. Both trends were an increase in G' with an increase in test temperature up to 14°F (-10°C) and then a decrease as the test temperature continued to increase.

The statistical analysis of the 72 hour accelerated cured, unaged sealant A material indicated that none of the exhibited differences were statistically significant. Therefore, all of the test data could be combined. The statistical analysis implied that G' did change with changes in temperature. The trend exhibited by the statistically combined data was different than the trend exhibited by the original data. The trend of the original data was an increase in G' as the test temperature increased up to a temperature of 32°F (0°C) and then a decrease as the temperature continued to increase.

The statistical analysis of the 21 day laboratory cured, unaged sealant A material indicated a somewhat less than logical combination of the data. For example, the 1 Hz data collected at -22°F (-30°C) was found to be statistically different than the -4°F (-20°C) through 50°F (10°C) data and the 104 and 122°F (40 and 50°C) data. However, the differences between the -22, -4, and 86°F (-30, 20, and 30°C) data were not statistically significant. If the -22°F (-30°C) data were eliminated from consideration, the remaining data combinations would be considered more logical, i.e., data with sequential test temperatures were combined. The trend exhibited by the statistically combined data was similar to the trend exhibited by the original data.

The statistical analysis of the 24 hour accelerated cured, 7 day oven aged sealant A material generally exhibited a logical combination (sequential test temperature combinations) of the data. The exception to the logical combination was the -22°F (-30°C) data. The analysis indicated that the -22°F (-30°C) data could

be combined with the 68, 86, and 104°F (20, 30, and 40°C) data but not with the -4, 14, 32, or 50°F (-20, -10, 0, or 10°C) data. The combination of the data did not change the exhibited trend of G' versus temperature. However, if the -22°F (-30°C) data were eliminated, the generalized trend of G' would be a decrease with increasing test temperatures.

The 48 hour accelerated cured, 7 day oven aged sealant A material statistical analysis indicated that the differences between all of the data, with the exception of the 50 and 68°F (10 and 20°C) data, were not statistically significant. The 50 and 68°F (10 and 20°C) data were statistically different from each other and from all of the other data. The trend exhibited by the combined data was no change in G' to 32°F (0°C), a decrease to 50°F (10°C), an increase to 68°F (20°C), a decrease to 86°F (30°C) and then no change as the test temperature continued to increase. This response did not seem logical when considered from a material characterization perspective.

The statistical analysis for the remaining conditions of sealant A were similar to the ones that have been discussed. Some data combinations were temperature sequential and some data combinations skip two or three of the intermediate temperatures but could be combined with non-sequential temperatures. Additionally, some of the trends of the combined data were slightly different than the original data while others did not change. It was suspected that variability in sample preparation, the testing procedures and in the material itself as well as the small sample population caused the unexpected statistical data combinations or the data combinations that did not correspond with the combinations that would be expected through material characterization considerations.

Sealant B Statistical Analysis

The statistical analysis of the sealant B samples which were exposed to the same curing and aging conditions and tested at the same temperature indicated that the differences between many of samples were statistically significant. Examining the graphical representation of the individual specimen data indicated that in general,

the two samples for test condition exhibited similar trends. Also the range of the data at each test temperature for each test condition did not appear to be as large as the range exhibited by the sealant A data. It should be noted that only two samples were tested at each condition; therefore, the variability between samples would normally be expected to be less when testing two samples from the same sheet of cured sealant versus samples taken from two or more sheets of cured material.

The fact that the differences were determined to be statistically different was expected. The data indicated that the three measurements for the majority of the individual samples were almost identical yielding standard deviations of either zero or a very small number as compared to the storage modulus. The differences exhibited in the samples of the remainder of the various testing conditions were generally determined to be statistically significant.

The statistical analysis of the temperature data within a specific curing and aging condition provided inconsistent results. For this portion of the statistical analysis, all of the data obtained at a specific temperature and test condition were averaged. The statistical analysis of the 24 hour accelerated cured, unaged sealant B material indicated that the differences in G' from -22 to 50°F (-30 to 10°C) and between the 68 and 86°F (20 and 30°C) data were not statistically significant. However, the differences between the combined -22 to 50°F (-30 to 10°C) and the 68 to 86°F (20 to 30°C) as well as the 104°F (40°C) data and 122°F (50°C) data were statistically significant. The combinations delineated by the statistical analysis were logical in that the data that were combined were temperature sequential. This analysis indicated that the G' response versus temperature could be divided into four general region; less than 50°F (10°C), between 68 and 86°F (20 and 30°C), 104°F (40°C), and 122°F (50°C). The trend exhibited by the combined data was the same as that exhibited by the original data. The exhibited trend was a decrease in G' with increases in test temperatures.

The data combinations delineated by the statistical analysis of the 48 hour accelerated cured, unaged sealant B material generally followed sequential test

temperature combinations. There were two exceptions to sequential test temperature combinations; the 50°F (10°C) data which was significantly different than the 14°F (-10°C) data but not significantly different from the -4 or -22°F (-20 or -30°C) data, and the -22°F (-30°C) data which was significantly different than the 14°F (-10°C) data but not significantly different than the -4, 32, or 50°F (-20, 0, or 10°C) data. Statistically combining the data did not alter the trend exhibited by the original data.

The 72 hour accelerated cured, unaged sealant B material statistical analysis indicated that the data could be combined into two groups; the -22 to 68°F (-30 to 20°C) data and the 86 to 122°F (30 to 50°C) data. The combination of the data followed sequential test temperatures and basically indicated that below 68°F (20°C), one G' value could be used and above 68°F (20°C) another single value for G' could be used. The generalized trend from the data combination was slightly different than the original data trend. The original data trend was an increase in G' with an increase in temperature to 32°F (0°C) and a decrease in G' as test temperatures continued to increase. The combined data trend was no change in G' to 68°F (20°C), a decrease between 68 and 86°F (20 and 30°C), and then no change in G' above 86°F (30°C).

The statistical analysis of the 24 hour accelerated cured, 7 day oven aged sealant B material indicated that the data could be divided into four general areas; -22°F (-30°C), -4°F (-20°C), 14 through 86°F (-10 through 30°C), and 104°F (40°C) and 122°F (50°C). However; one non-sequential temperature data combination did exist and it was the -4°F (-20°C) data combined with the 104 and 122°F (40 and 50°C) data. The trend of the combined data was similar to the trend exhibited by the original data.

Only the 50 through 122°F (10 through 50°C) data were available for the 48 hour accelerated cured, 7 day oven aged sealant B material. The statistical analysis of the available data indicated that the 50 through 104°F (10 through 40°C) data could be statistically combined but the differences between the 50 through 104°F (10 through 40°C) data and the 122°F (50°C) data were statistically significant. The

trend exhibited by the combined data was no change in G' from 50 to 104°F (10 to 40°C) and then a decrease in G' at 122°F (50°C). The original trend had been a decrease in G' with increases in temperature from 50 to 122°F (10 to 50°C).

The 86 through 122°F (30 through 50°C) data were not available for the 72 hour accelerated cured, 7 day oven aged material. The statistical analysis of the remaining data indicated that all of the data could be combined with the exception of the -4°F (-20°C) data. However, the 68°F (20°C) G' value was approximately twice that of the combined average of the remaining data indicating that it was a potential outlier. The trend of the combined data was basically no change in G' with an increase in test temperature whereas the trend for the original data was generally a decrease in G' with an increase in test temperature.

The remaining sealant B data indicated similar trends to those that have been discussed, i.e., some of the test conditions had non-sequential test temperatures that could be statistically combined while other test condition combinations were sequential. The overall effect of the data combination generally caused no change from the original data trend or changed the data trend to a constant G' with changes in temperature instead of an increase or decrease with changes in test temperature. There was no consistent pattern to the combination of non-sequential test temperatures. For example, if the -22°F (-30°C) data could consistently be combined with non-sequential test temperatures (i.e., 86 or 104°F (30 or 40°C)), then the lower G' value obtained at -22°F (-30°C) could have been caused by slippage between the test specimen and the parallel plates. However, no such consistency existed.

Statistical Analysis of the Remaining Sealants

The statistical analysis of sealants C, D, E, and F exhibited the same inconsistent results. Generally, the differences between the individual samples tested at the same curing and aging conditions were statistically significant. There were some instances where the differences between the individual samples were determined to not be statistically significant but these were isolated occurrences.

The differences between the test temperatures at the various conditions were also similar to sealants A and B in their inconsistency. Some data in which the differences were determined to not be statistically significant were illogical combinations while others followed a more logical pattern of temperature combinations.

Statistical Analysis of G' Versus Temperature

A final area investigated on the initial data was an attempt to determine if the G' versus temperature trends exhibited by two individual samples tested at a given test condition were similar. Even though differences in the test results were statistically significant, this analysis could indicate that trends associated with increases or decreases in G' with respect to temperature could occur in both samples. The results from this analysis were as inconsistent as the results from the previous analysis. In other words, some of the individual samples tested at the same conditions exhibited an almost identical trend when comparing G' versus temperature but other samples exhibited no similarities.

One problem with the initial statistical analysis was the small sample population, only two samples at each test condition. In an attempt to address this problem, additional samples of sealant F were prepared and tested. The test conditions used for the additional samples were 24 hour accelerated cure, 24 hour accelerated cure with 24 hours of PAV aging, 21 day laboratory cure, and 21 day laboratory cure with 7 days of oven aging. The additional test results when included with the initial test results did not greatly reduce the coefficients of variation. However, the G' versus temperature trends did become more similar to the expected trend (i.e., decreased G' with increased temperature). There was only enough material for an additional four to eight samples and it was believed that to accurately determine statistical significance a greater number of samples would have to be tested. Since the coefficients of variation were slightly reduced with the inclusion of the additional samples, only sealant F was used to develop and calibrate the numerical model.

DSR Testing Variability

Statistical analysis of the initial dynamic shear rheometer data of the sealant characterizations indicated coefficients of variation ranging from approximately 1 to 78 percent. The range exhibited by the coefficients of variation were larger than expected. However, the majority of coefficients of variation were less than 20 to 30 percent. One could consider coefficients of variation in the 20 to 30 percent range too high to allow accurate field performance predictions. Before the results were considered "no good," the accepted testing accuracy used in the paving industry and potential causes of the variability were evaluated.

Silicone materials were moisture curing systems; therefore, it was expected that the greatest variation would be exhibited in the samples with the least amount of cure time. As both cure time and length of aging was increased, it was expected that the coefficients of variation would decrease. A second potential cause of variation was the fact that the operator was not familiar with preparing samples for DSR testing and was not familiar with the DSR testing techniques. The variability that would have resulted due to this potential cause should have been greater during the first few tests and then decreased as the testing continued. However, as previously discussed, the variability exhibited in the data did not follow either of these trends. Instead, the variability was random.

The majority of the coefficients of variation were less than 20 to 30 percent. Typically, a coefficient of variation of less than 20 to 30 percent was generally considered to be very good for the evaluation of engineering materials especially in the pavement industry, as discussed in Chapter 2. The seemingly high coefficients of variation generally obtained during civil engineering material characterization testing were typically attributed to the non-uniformity of the material being tested and the small sample population used to characterize the material.

The coefficients of variation exhibited in the DSR sealant characterization, although higher than would normally be desired for performance prediction, was within values typically obtained from other material characterization techniques used

by the paving industry. The coefficients of variation obtained from the DSR sealant results are also generally lower than those exhibited by other industry accepted joint sealant testing techniques. Therefore, the use of DSR to characterize joint sealant materials provided a more repeatable method to characterize sealants in addition to providing information more directly related to field performance.

Poisson's Ratio Results

The average υ of three of the sealant materials are provided in Table 4.19. There was not enough of the other three materials to pour samples to allow υ to be calculated. In previous research conducted on silicone sealant materials, the υ was generally assumed to be 0.49 or 0.5. One exception to this trend was the work conducted by Wang [47]. Wang measured υ for one silicone material to be approximately 0.471.

The average υ measured for the three sealants ranged from 0.41 to 0.47 with coefficients of variation ranging from 15 to 30 percent. The calculated standard deviations for the measurements used to determine the υ would make it numerical possible for values to be obtained that were above 0.5. Incompressible materials have a maximum υ of 0.5 and υ values above 0.5 are not realistic for traditional materials, and, therefore, values above 0.5 were eliminated. The values for υ listed in Table 4.19 were used in the numerical modeling input. A sensitivity analysis of υ was conducted in the numerical analysis because of the variability exhibited with the measurements.

Table 4.19. Average Poisson's Ratio for Selected Silicone Sealants.

SEALANT	AVERAGE POISSON'S RATIO	COEFFICIENT OF VARIATION
Sealant D	0.41	30%
Sealant E	0.41	17%
Sealant F	0.47	15%

Tensile Test Results

Sealant F was selected for tensile testing to be used to verify the numerical model. The main reason for the selection of sealant F was the fact that the shelf life for the other materials obtained for testing had expired or was close to expiration.

The results from the tensile testing are provided in Table 4.20. The thicknesses referred to in Table 4.20 was the thickness from the top surface of the sealant to the top of the parabolic surface on the bottom of the sealant. The average results provided for each sample were three repeated tests on the material with the exception of the 25 percent elongation results. The coefficient of variation for the majority of laboratory tests conducted on the silicone sealants was less than 25 to 30 percent. Therefore, a coefficient of variation of 25 percent was assumed for the 25 percent elongation data and a 95 percent confidence region ($\propto = 0.5$) was used to determine if any of the values could be considered an outlier. For the 0.25 inch (6.35 mm) thick material, one value could be deleted as an outlier. The deletion changed the mean load per unit length from 1.33 lbs (0.60 kg) and a coefficient of variation of 56 percent to 0.90 lbs (0.41 kg) with a coefficient of variation of approximately 18 percent. The assumed coefficient of variation of 25 percent also delineated one value of the 25 percent elongation data for the 0.125 inch (3.18 mm) thick sample as a potential outlier. The deletion of the potential outlier from the 0.125 inch thick sample changed the mean value at 25 percent elongation from 0.76 lbs (0.34 kg) and a coefficient of variation of 51 percent to 0.53 lbs (0.24 kg) with a coefficient of variation of approximately 7 percent. None of the other reported values could be eliminated using this criteria.

After the load at 100 percent elongation had been recorded the third time, the samples were extended in excess of 500 percent elongation and the sealant was cut through the center. The cutting of the sealant was done to determine if voids were present in the material. None were present indicating that the results obtained from the tensile test should be representative of the material property.

Table 4.20. Tensile Test Results For Sealant F.

Elongation	Sample 1 (0.25 inch Thickness)		Sample 2 (0.125 inch Thickness)	
	Average (Pounds per Linear Inch)	Coefficient of Variation	Average (Pounds per Linear Inch)	Coefficient of Variation
25%	0.90	17.5	0.53	7.4
50%	1.51	21.5	1.13	23.8
75%	1.70	11.1	1.39	13.8
100%	1.94	5.2	1.61	7.6

CHAPTER 5 - TESTING VARIABILITY OF
CIVIL ENGINEERING MATERIALS

Variability in DSR Characterization

The variability associated with the material characterization of civil engineering materials was discussed in Chapters 2 and 4. A summary of those two discussions are presented in this chapter because the review of current practice will delineate the improvements and advancements made in joint sealant characterization.

A brief statistical analysis of the initial dynamic shear rheometer data of the sealant characterizations indicated that the coefficients of variation ranged from approximately 1 to 78 percent. The range exhibited by the coefficients of variation was larger than expected. The main reason smaller coefficients of variation were expected was that most literature that presented shear modulus data did not present variability data. Therefore, one would potentially assume that the error was very small. Additionally, when performance-based specifications were developed from the data, as in the Strategic Highway Research Program for asphalt cements, the potential assumption would be that the variability in test results was probably less than 5 percent. However, the approximate coefficient of variation for asphalt cement characterization using the dynamic shear rheometer was approximately 34 percent [44]. The majority of the coefficients of variation for the DSR joint sealant characterization were less than 20 to 30 percent. Therefore, comparing the variability of the joint sealant characterization results to the variability of the asphalt cement characterization, indicated that the variability was not unreasonable.

Typically, a coefficient of variation of less than 20 to 30 percent was generally considered to be very good for the evaluation of engineering materials especially in the pavement industry. The seemingly high coefficients of variation generally obtained during civil engineering material characterization testing are attributed to the non-uniformity of the material being tested and the small sample population generally used to characterize the material.

Potential Causes of Silicone Material Variability

The silicone materials used in this investigation were moisture curing systems; therefore, it was expected that the greatest variation would be exhibited in the

samples with the least amount of cure time. As both cure time and length of aging was increased, it was expected that the coefficients of variation would decrease. A second potential cause of variation was the fact that the operator was not familiar with preparing samples for DSR testing and was not familiar with the DSR testing techniques. The variability that would have resulted due to this potential cause should have been greater during the first few tests and then decreased as the testing continued. However, as discussed in Chapter 4, the variability exhibited in the data did not follow either of these trends. Instead, the variability was random.

Variability Exhibited in Current Sealant Specifications

The current joint sealant specification tests used for hot-applied joint sealant materials exhibited a wide range of coefficients of variation. A recent study conducted jointly by WES and Crafco, Incorporated, [25] indicated that the coefficient of variation for the unaged initial indentation test (a test procedure similar to a penetration test) ranged from approximately 9 to 64 percent. The coefficient of variation for the same test on aged joint sealant materials ranged from 0 to 69 percent. The coefficients of variation for the other tests such as flow, penetration, and resilience ranged from approximately 1 to 177 percent. The potential reasons of the large variation of these test procedures were variability of the sealant materials and variability of the test procedures. Results of the WES/Crafco study delineated the need for round robin testing to allow precision and bias statements to be developed for the ASTM sealant specifications. The round robin testing indicated the variation exhibited in the test methods were due in part to the type of material being tested. For example, the results of two properly conducted penetration tests in different laboratories of a hot-applied material which exhibited a penetration between 50 and 70 should not differ by more than 15 penetration units. The results of two properly conducted penetration tests in different laboratories of a hot-applied material which exhibited a penetration between 71 and 85 should not differ by more than 48 penetration units. The penetration requirement for Federal Specification SS-S-1401C was a maximum of 9

mm or 90 penetration units. The amount of variation implied that a material which in fact complied with the material specification requirements could yield test results which indicated non-compliance. The single operator precision for resilience testing was 3 units and the multi-laboratory precision was 33 units, again indicating that a material that actually conformed to specification requirements may be reported as non-compliant. An industry accepted specification does not exist for silicone sealants; therefore, precision and bias data were not available for the procedures used to test silicone sealants. However, variability similar to that exhibited in the other types of sealant specifications would be expected.

Variability in Other Civil Engineering Material Characterization Techniques

Another area of civil engineering material characterization that had exhibited a great deal of variability was soils testing. In the 1960's, a round robin was conducted which included approximately 100 of the "top" testing laboratories [45]. Three different soil materials; a low plasticity Vicksburg loess, a medium plasticity lean clay, and a high plasticity buckshot clay, were sent to the testing laboratories. The tests conducted on these soils were liquid limit, plastic limit, grain size, specific gravity, and standard and modified moisture-density relations. The basic conclusion of the study was that the variability of the routine tests was "...of considerable magnitude in many instances." The variation in testing was further delineated by the fact that the 95 percent confident region for the specific gravity of the high plasticity buckshot clay ranged from a minimum of approximately 2.4 to a maximum of approximately 2.9. This specific gravity range included almost every type of soil that has ever been evaluated.

Another material characterization technique was aggregate sieve analysis. In this procedure, an aggregate sample would be obtained and separated based on particle size using a series of sieves. One specification typically used for conducting a sieve analysis is ASTM C 136. The precision estimate for the coarse aggregate analysis listed in the precision and bias statement of the specification indicated that the coefficient of variation ranged from approximately 30 to 35 percent. The

variation was attributed to sampling error, the use of a non-representative sample, overcharging the sieves, and/or equipment and procedure errors, i.e., the condition of the equipment or the duration of aggregate sieving [46].

This brief review indicated that the coefficients of variation exhibited in the DSR sealant characterization, although higher than would normally be desired for performance prediction, was within values typically obtained from other material characterization techniques used by the paving industry. The coefficients of variation obtained from the DSR sealant results were also generally lower than those exhibited by other industry accepted joint sealant testing techniques. Therefore, the use of DSR to characterize joint sealant materials would provide a more repeatable method to characterize sealants in addition to providing information more directly related to field performance.

CHAPTER 6 - NUMERICAL ANALYSIS

Model Development for Numerical Analysis

The finite element code used for the numerical modeling of the joint sealant material in the joint was ABAQUS which was a general purpose, non-linear, transient dynamic finite element code. ABAQUS was selected because it has been validated extensively and it was readily available. The geometric mesh representing the joint sealant in the joint was generated using PDA Engineering's PATRAN code which included an ABAQUS application interface. The ABAQUS input files consisting of the generated mesh, material properties and loading conditions, were then submitted to one of the WES supercomputers (the CRAY Y-MP or the CRAY C-90) for analysis. Pre- and post-processing were conducted on a workstation.

The three-dimensional joint sealant was idealized by assuming plane strain conditions. In plane strain analysis, all strains are assumed to occur in the plane containing the cross-section of the sealant with no out-of-plane deformations. This was a valid assumption for the joint sealant because generally the configuration of the sealant in the joint would be 0.5 to 1 inch (12.7 to 25 mm) wide by 0.5 to 1 inch (12.7 to 25 mm) deep by 15 feet (4.57 m) or more in length.

Two different geometric models were used in the numerical analysis. The first geometry was the sealant only and it was selected for analysis because it was a simple representation of the sealant for characterization purposes. The second geometry included the concrete "slab." The second geometry was used for the majority of the analysis because it was more representative of field conditions and provided information concerning the sealant/concrete interface. Both geometries incorporated boundary conditions such that the left and right sides of the seal could not move in the y direction and a time dependent deformation in the x direction was induced on both the right and left sides. These boundary conditions would be similar to those that the sealant would experience in the field. The dimensions used for the sealant geometry were measured from a field sample taken from Naval Air Station Key West. This configuration was representative of the in-place sealant configuration recommended by most of the silicone sealant manufacturers. A

representation of the two geometries used in the analysis along with the triangular elements and aspect ratios of the elements is provided in Figures 6.1 and 6.2.

In the sealant/concrete numerical analysis, the concrete was represented by two elastic platens which were given material properties similar to that of a concrete pavement. The input properties were an elastic modulus of 4,000,000 psi (27,576 MPa) and a Poisson's ratio of 0.2. The sealant/concrete interface was modeled to have 100 percent adhesion (i.e., the sealant could not exhibit adhesion failure during the modeling process).

The first attempts of analysis modeled the sealant as an elastic material using first order triangular elements and then first order quadrilateral elements. Both of the first order element models calculated a force per unit length that was approximately 20 percent less than the force per unit length measured in the tensile testing. The first order element models also could not model deformations greater than approximately 30 percent elongation. The sealant was then modeled as a viscoelastic material again using first order triangular and quadrilateral elements. The amount of elongation in the viscoelastic model was greater than 30 percent but elongations greater than approximately 40 percent could not be achieved.

In typical field applications, the pavement joint should be designed in such a manner that the sealant would not exposed to movements greater than approximately 25 percent. However, many specifications have included laboratory tests that require elongation of the sealant to 100 or 200 percent. Therefore, an attempt was made to include the larger elongations in the numerical analysis. Higher order elements were used in the mesh as one potential method to eliminate the numerical instability at elongations greater than 30 percent. The use of higher order elements did allow larger elongations of the sealant than 30 percent but the full range of elongations (up to 100 percent) could not be obtained. A second attempt to correct the difficulties with the numerical model, included reducing the time step from 10 seconds to 1 second. This change reduced the amount of time that the analysis tried to model in each interval and it did increase the amount of elongation to above 50 percent.

Aspect Ratio

5.03	
5.00	
4.72	
4.43	
4.15	
3.87	
3.58	
3.30	
3.02	
2.73	
2.45	
2.17	
1.88	
1.60	
1.32	
1.03	
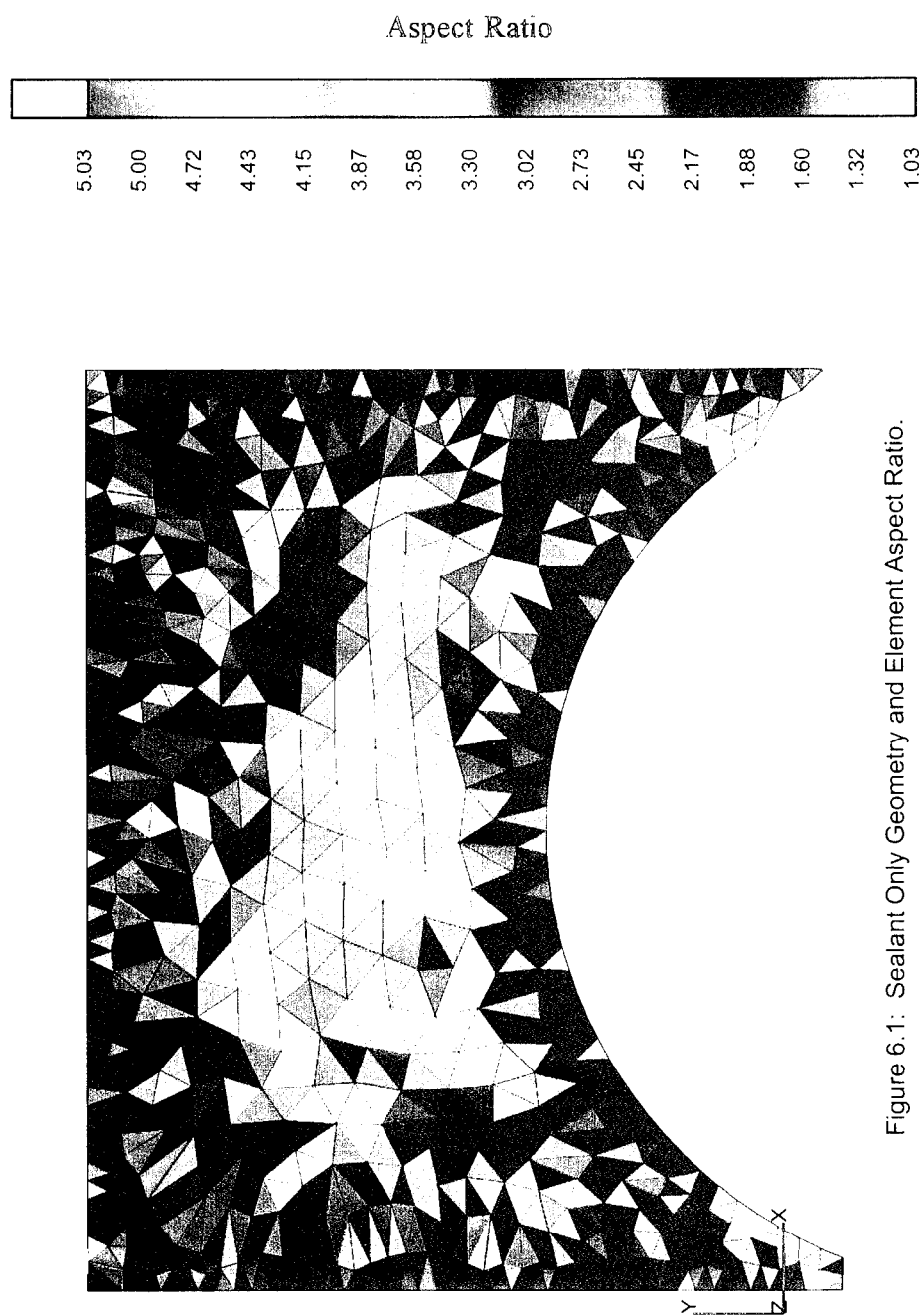
Figure 6.1: Sealant Only Geometry and Element Aspect Ratio.

Aspect Ratio

24.3
5.00
4.71
4.43
4.14
3.86
3.57
3.29
3.00
2.72
2.43
2.14
1.86
1.57
1.29
1.00
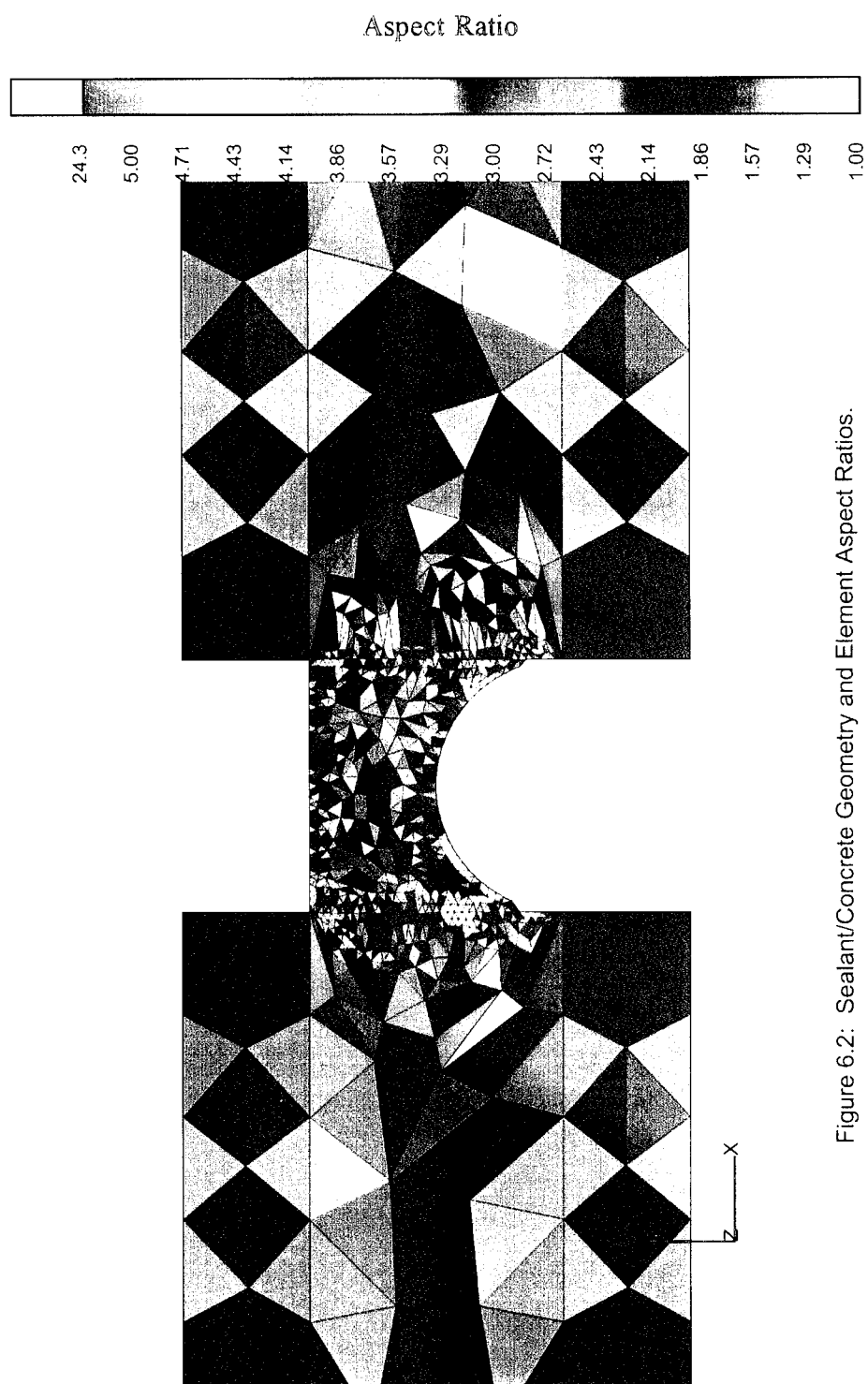
Figure 6.2: Sealant/Concrete Geometry and Element Aspect Ratios.

However, the deformation exhibited by the model at approximately 50 percent elongation was not representative of the deformation exhibited in the laboratory tested samples. In an effort to correct this problem, the number of elements in the sealant were more than doubled. Several of the elements at the bottom of the sealant/concrete interface had to be deleted because they had angles of less than 45 degrees which created a problem in the numerical analysis. Increasing the number of elements in the sealant section did not correct the inaccurate deformation problem and it created additional numerical stability problems in the model.

Since none of the previous models remained numerically stable at elongations above approximately 30 to 50 percent an input value called CETOL was added to the model. This input value helped to reduce numerical instability by allowing non-exact solutions to be acceptable during the various numerical iterations. Normally, the numerical calculations must be exactly equal at each iteration or the analysis would be terminated. The CETOL provided a tolerance around the solution to prevent or reduce the probability of analysis termination. This model was capable of achieving elongations of up to 100 percent and the model was reconstructed using the first order triangular elements. The next step in the modeling process was to reduce the CETOL value to the minimum value that would still allow the analysis to complete result calculations.

Viscoelasticity in the Numerical Analysis

The viscoelastic material model used to represent the material behavior was a Prony series. The Prony series representation of the shear modulus is [55]:

$$g_R(t) = \frac{G_r(t)}{G_0} = 1 - \sum_{i=1}^{N} g_i^P (1 - e^{-t/\lambda_i}) \tag{24}$$

where G_r is the shear relaxation modulus, g_i^P were the modulus ratios in the Prony series expansion, and G_0 is the instantaneous shear modulus. For a solid material

$$G_0 = G_\infty + \sum_{i=1}^{N} g_i \qquad (25)$$

where G_∞ is the shear modulus at infinity and g_i are weighted factors corresponding to the relaxation times calculated from the IRIS software. Finally, the last step required to obtain the ABAQUS input was converting from g_i to g_i^P. This conversion was made by:

$$g_i^P = \frac{g_i}{G_0} \qquad (26)$$

The information obtained or derived from the DSR testing required for the numerical analysis was G_∞, g_i^P, and λ_i. The mechanics for obtaining this information was relatively straight forward. The first step was to develop a mastercurve from the individual DRS test data. The G_∞ was then estimated from the rubbery plateau region of the mastercurve as the minimum value of G'. The true value for G_∞ would not be easy to determine because the length of testing would be very long; however, the estimation could be made because for a solid material, the measured G' from the oscillatory experiment would be the sum of the G_∞ and the transitional storage modulus (G'_{trans}).

The minimum value of G' (which becomes G_∞) was subtracted from the remaining G' values and the discrete relaxation spectrum was calculated using the modified G' and the original G''. The discrete stress relaxation spectrum provided g_i and λ_i. The g_i values were then added to G_∞ to calculate G_0 and the g_i^P values were calculated using g_i and G_0. These calculated values along with λ_i and υ were used as the direct input to the finite element code.

Model Boundary Conditions

The joint movement first used in the model was the same as used in the laboratory testing and was accomplished by displacing both concrete platens at a displacement rate of 0.125 inches (3.18 mm) per minute to a total displacement of

0.5 inch (12.7 mm) or 100 percent elongation. The 100 percent elongation was larger than would typically be expected in the field, but it was a standard amount of elongation used for laboratory testing of joint sealant materials. The rate of displacement used in the boundary conditions was also higher than would be expected in the field for movements caused by temperature and/or moisture changes in the slab. This rate was selected for tensile testing because it was the typical rate used for bond testing in many of the joint sealant material specifications and it was convenient and expedient for laboratory testing.

Once the model had been verified using the laboratory test conditions and results, more realistic field conditions were used as input into the model for analysis. The displacements used for field simulations were accomplished by moving both edges of the sealant at a displacement rate of 0.125 inch (3.18 mm) per hour to a total displacement of 0.25 inch (6.35 mm) or 50 percent elongation. Additionally, some numerical analysis were made by displacing one of the concrete blocks vertically 0.25 inch (6.35 mm) within one second and then returning the concrete block to the original position. This type of movement was used to simulate faulting of a pavement.

Mesh Sensitivity Analysis

There were two areas of the numerical analysis that required additional investigation to determine the sensitivity of the analysis to those factors. The first factor was the sensitivity of the analysis to the mesh used. It was important to verify that the results that were obtained from the numerical analysis were not specifically due to the type of element selected and to the coarseness of the mesh. The verification or calibration of the model to laboratory test data greatly assisted in the sensitivity analysis in that if the numerical analysis yielded similar results to the laboratory testing, then one would be confident in the model. However, if laboratory data or some other means of verification were not available, the sensitivity analysis would become much more critical.

Triangular Elements Versus Quadrilateral Elements

The mesh sensitivity was analyzed by first comparing the results obtained by using first order quadrilateral elements (approximately 700 elements were used in the quadrilateral model) to first order triangular elements (approximately 800 elements were used in the triangular model). Both of these models calculated a force per unit length that was within approximately 2 percent of the force per unit length determined through laboratory tensile testing. It appeared from these results that either a quadrilateral or triangular element could be used to represent the sealant material. However, attempts to increase the number of elements using the quadrilateral elements in the model created some difficulty because of the geometry of the sealant. Therefore, the triangular element was determined to be more appropriate for the sealant modeling.

Number of Elements

After the selection of the triangular element for the model, the mesh sensitivity analysis focused on the number of elements or fineness of the mesh. The mesh sensitivity was determined by incrementally increasing the number of elements and calculating the resultant force per unit length at 25 percent elongation. The calculated force per unit length was then compared to the previously calculated result and/or laboratory data. The steps of increasing the number of elements and evaluating the results continued until the difference between two calculated results was within a determined satisfactory range or was within a determined satisfactory range of the laboratory data. Very often laboratory data would not be available for the analysis, and, therefore, comparisons between two calculated results would become the deciding factor in determining if enough elements had been incorporated into the model. Another deciding factor in determining the number of elements that could or should be used in a model would be the practicality of running the analysis. It may not be important to be within less than 1 percent of the laboratory results or the previously calculated value if the model required two weeks of computing time to complete.

The results of the mesh sensitivity analysis indicated a significant difference based upon the total number of elements selected to represent the joint sealant material. Table 6.1 provides the summarized results from the sensitivity analysis using the sealant F dynamic shear rheometer data obtained at reference temperature of 68°F (20°C) after 21 days of laboratory curing and a υ of 0.47. These results indicated that as the number of elements increased, the resultant force per unit length calculated by the model became closer to the force per unit length measured in the laboratory tensile testing. When 236, three node triangular elements were used, the resultant force per unit length calculated by the numerical was approximately 43 percent higher than the values measured in the laboratory. The deviation between the laboratory tensile test results and the numerical analysis decreased significantly to 6 percent higher than the laboratory results when the number of elements was increased to 487. The deviation continued to decrease as the number of elements were increased to in excess of 1,000. The deviation between the laboratory test and the this last model was approximately 0.4 percent.

Table 6.1 Mesh Sensitivity Analysis for Sealant F at 25 Percent Elongation Using Three Node Triangular Elements.

Number of Elements	Calculated Force Per Unit Length (lbs.)	Percent of Laboratory Results
236	1.290	43
487	0.959	6
>1,000	0.897	<1

The sealant only model was reconstructed using six node triangular elements to determine if the deviation between the calculated results and the laboratory results could be further reduced. This step was included because, in general, higher

order elements are often believed to produce more accurate results. Table 6.2 provides the results from the six node triangular element model analysis. The results from the six node triangular model were less stiff as would be expected; however, even with the incorporation of over 3,000 elements in the model, the deviation between the laboratory obtained results and the model calculated results was approximately 12 percent. Based on this analysis the three node triangular element model with approximately 1,000 elements was used for the remainder of the analysis.

A second verification of the three node, 1,000 plus element model was made at 50 percent elongation. At 50 percent elongation the deviation between the model calculated and the laboratory determined results was approximately 18 percent with the model calculated values being lower than the laboratory obtained values. The model verification at 25 and 50 percent indicate that for elongations of approximately 25 percent the model will very accurately characterize the sealant. As the amount of elongation increases, the model becomes less accurate in the sealant characterization. Additional refinement to the numerical model will be required if larger elongations are to be modeled accurately.

Table 6.2 Mesh Sensitivity Analysis for Sealant F at 25 Percent Elongation Using Six Node Triangular Elements.

Number of Elements	Calculated Force Per Unit Length (lbs.)	Percent of Laboratory Results
275	0.689	24
694	0.709	21
1026	0.793	12
3,298	0.793	12

The above mesh sensitivity analysis was conducted on a model representing the sealant tensile samples which were 0.25 inch (6.35 mm) thick in the center of the sealant cross-section as described in Chapter 4. A second model was generated to represent the sealant tensile samples which measured 0.125 inch (0.318 mm) thick in the center of the sealant cross-section. Only one mesh containing 603 elements was used with this geometry. The results calculated using the 603 element model calculated a force per unit length of 0.475 pounds which was approximately 11 percent less than the force per unit length determined in the laboratory tensile testing. The deviation between the laboratory obtained and the model calculated results was larger than exhibited by the 0.25 inch (6.35 mm) thick model, but still within a satisfactory range. The deviation could probably be reduced by increasing the number of elements used in the mesh. The similarities between the results calculated by the two numerical models (0.25 inch thick and 0.125 inch thick) as compared to the laboratory obtained results indicated the validity of the model. Additionally, it demonstrated that the results were dependent on the fineness of the mesh. The similarities also indicated that even though the coefficients of variability for the DSR testing, the υ calculations, and the laboratory tensile testing were larger than desired, the average of those results provided a satisfactory characterization of the material properties. The results also indicated that the methodology developed to characterize the sealant materials was valid.

Model Sensitivity to Poisson's Ratio

A second area that required investigation was the sensitivity of the numerical model to the value of Poisson's ratio (υ). The ranges for υ that were calculated for three of the sealant materials was relatively large. It could be possible that the results calculated in the numerical model would be lower than the values determined by laboratory tensile testing because the value calculated for υ was in fact lower than the actual material value. It was therefore important to determine the significance of changes in υ to the results predicted from the model.

The sensitivity of the analysis to υ was determined by simply changing the value of υ and the corresponding elastic modulus in the input. The analysis was conducted and the resultant force per unit length compared to the values calculated over a range of υ. The sealant/concrete geometry was used for this analysis. Four values of υ were used for the three node triangular element analysis; 0.40, 0.45, 0.47, and 0.49, and three values were used for the six node triangular element analysis; 0.45, 0.47, and 0.49. Tables 6.3 and 6.4 provide the summarized data from this analysis.

The effects of changes in υ were greater for the three node triangular model than the six node triangular mode, but in both models, the number of elements in the model or mesh fineness affected the calculated results much more than changes in υ. It was interesting to note that the calculated force per unit length for the three node triangular model and a υ of 0.47 was within approximately 0.4 percent of the laboratory obtained values. These results provided additional confidence in the model and the fact that the average test results obtained from laboratory evaluation sufficiently characterized the sealant material. It also delineated the need to determine the exact number of samples that must be tested to ensure a representative average.

Table 6.3 Numerical Model Sensitivity to Changes in Poisson's Ratio Using Three Node Triangular Elements.

Poisson' Ratio	Calculated Force Per Unit Length (lbs.)	Percent of Laboratory Results
0.40	0.842	6
0.45	0.855	5
0.47	0.897	<1
0.49	0.976	8

Table 6.4 Numerical Model Sensitivity to Changes in Poisson's Ratio Using Six Node Triangular Elements.

Poisson' Ratio	Calculated Force Per Unit Length (lbs.)	Percent of Laboratory Results
0.45	0.790	12.3
0.47	0.793	11.9
0.49	0.796	11.7

Field Simulation Numerical Analysis Results

All of the numerical models (the sealant only and the sealant plus concrete), exhibited non-realistic deformations at elongations above approximately 55 to 75 percent. Typically, the sealant only models exhibited deformations similar to those observed in the laboratory testing up to approximately 75 percent elongation. At approximately 75 percent elongation, the elements in the lower center portion of the cross-section began to "collapse." The non-realistic deformation could have been caused by the fact that the aspect ratio of those elements were well in excess of 5, the fact that the CETOL allowed residual stresses to accumulate during the numerical analysis, or a combination of both. In the concrete/sealant models, the non-realistic deformation began to occur at approximately 55 percent. Figures 6.3 through 6.5 illustrate the calculated maximum principal stresses in the sealant/concrete model at elongations of 25 percent, 50 percent, and 75 percent elongation. In Figure 6.4, the non-realistic deformation has initiated and in Figure 6.5, the "collapse" has occurred.

Additional refinement to the model could have been attempted to minimize or eliminate the non-realistic deformation and to reduce the difference between the calculated and laboratory determined force per unit length of the sealant/concrete model at the higher elongations, but the objective of this research was to develop a

methodology using dynamic shear rheometry that would more adequately characterize silicone pavement joint sealant materials. The numerical analysis comparisons to the laboratory tensile testing verified that the averaged dynamic shear rheometer data provided an accurate methodology for the characterization of silicone sealant material properties.

The maximum principal stresses calculated in the field simulation numerical model at 25 percent elongation were very similar to the stresses calculated in the numerical model which used the higher displacement rate. Figure 6.6 provides an illustration of the calculated stresses in sealant F at 25 percent elongation. The calculated force per unit length at 25 percent for this model was 0.872 pounds per unit length or approximately 3 percent of the laboratory tensile test results. These results were very similar to the force per unit length calculated at the higher displacement rate of the sealant/concrete model. The DSR data exhibited a very gradual decrease or no change as the test temperature increased (i.e., as the rate of loading decreased). Therefore, the results between the two displacement rates should have been similar.

A second difference between the conditions used in the laboratory tensile testing and the conditions that would be experienced in the field was that instead of being simply extended, the sealant would be extended at a slow displacement rate, remain extended at some displacement (usually 25 to 50 percent elongation), and then compressed at a slow displacement rate to approximately the original width. This cycling could take hours or days to complete. For the purposes of this investigation, only the first half of the cycle was modeled. The representation of the extension to 25 percent elongation in the numerical model is illustrated in Figure 6.6. Figure 6.7 illustrates the stresses in the sealant at 25 percent elongation after approximately one hour of relaxation. There was no appreciable amount of stress relaxation after one hour. The lack of significant relaxation was expected due to the dynamic shear rheometer test results.

Maximum Principal Stresses (psi)

Figure 6.3: Sealant/Concrete Numerical Model Geometry at 25 Percent Elongation.

Maximum Principal Stresses (psi)

26.6
24.1
21.7
19.2
16.7
14.2
11.7
9.26
6.78
4.30
1.82
.656
-3.14
5.62
-8.10
-10.6
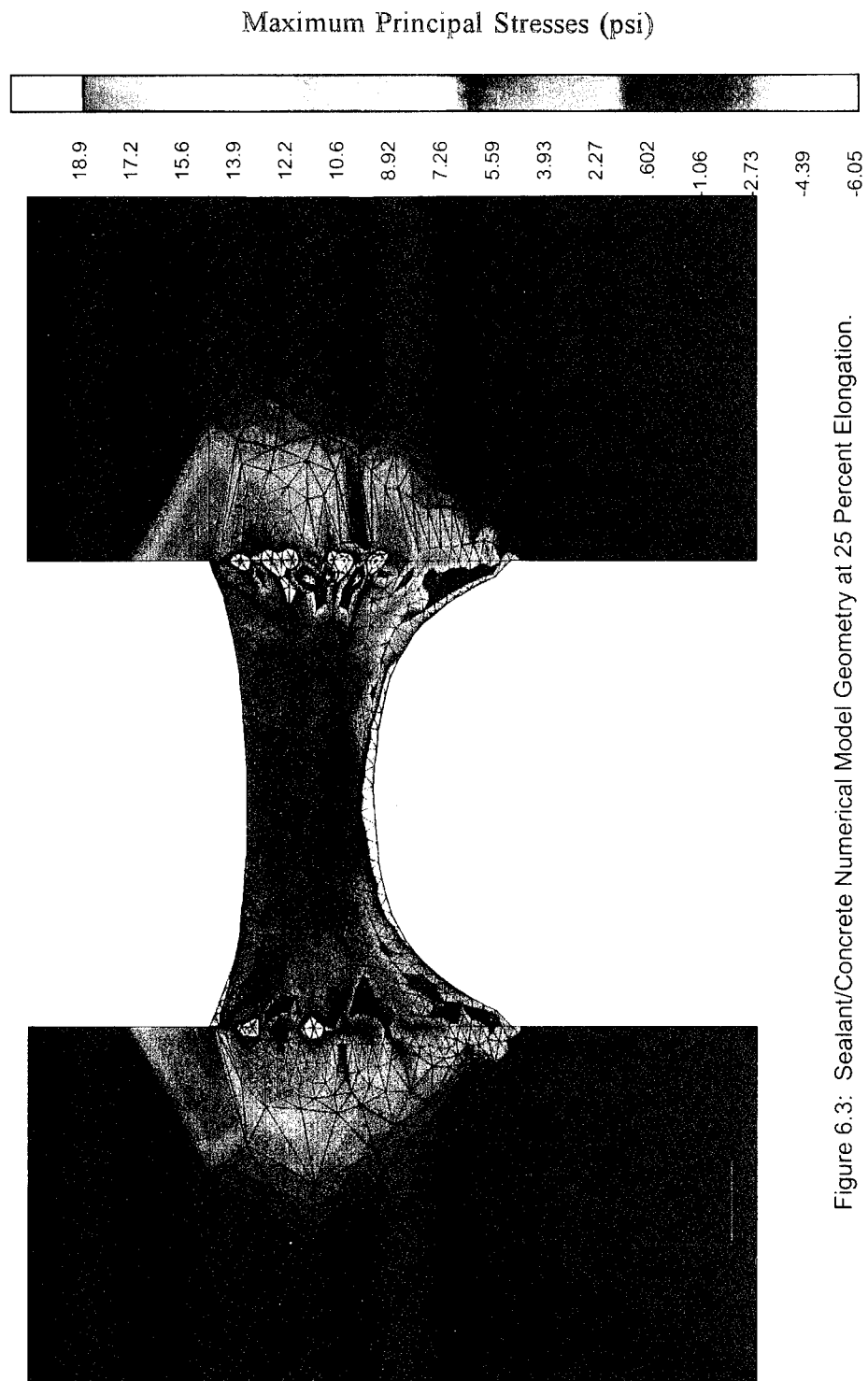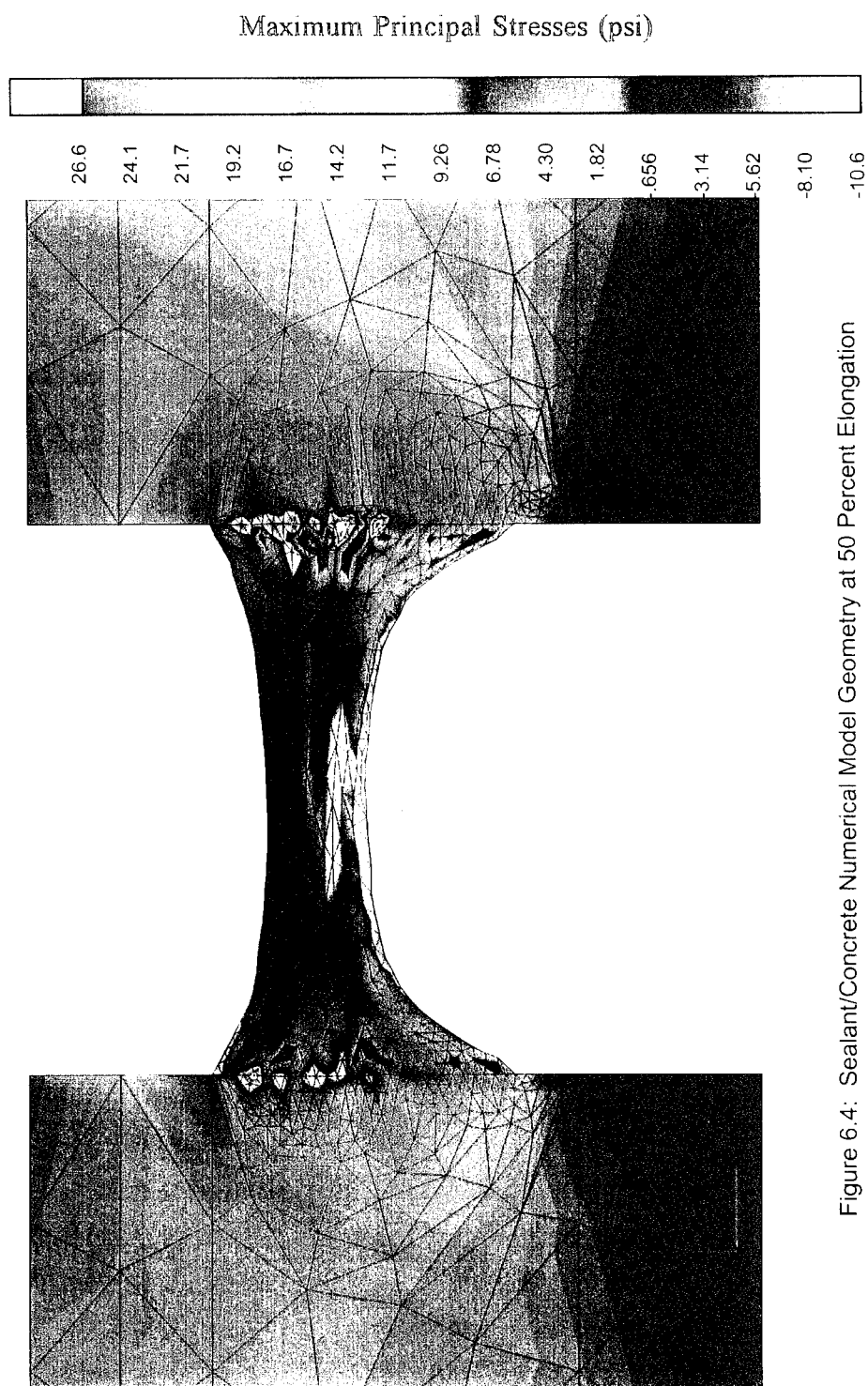
Figure 6.4: Sealant/Concrete Numerical Model Geometry at 50 Percent Elongation

Maximum Principal Stresses (psi)

19.9 17.7 15.5 13.4 11.2 9.05 6.89 4.73 2.56 .401 -1.76 -3.93 -6.09 -8.25 -10.4 -12.6
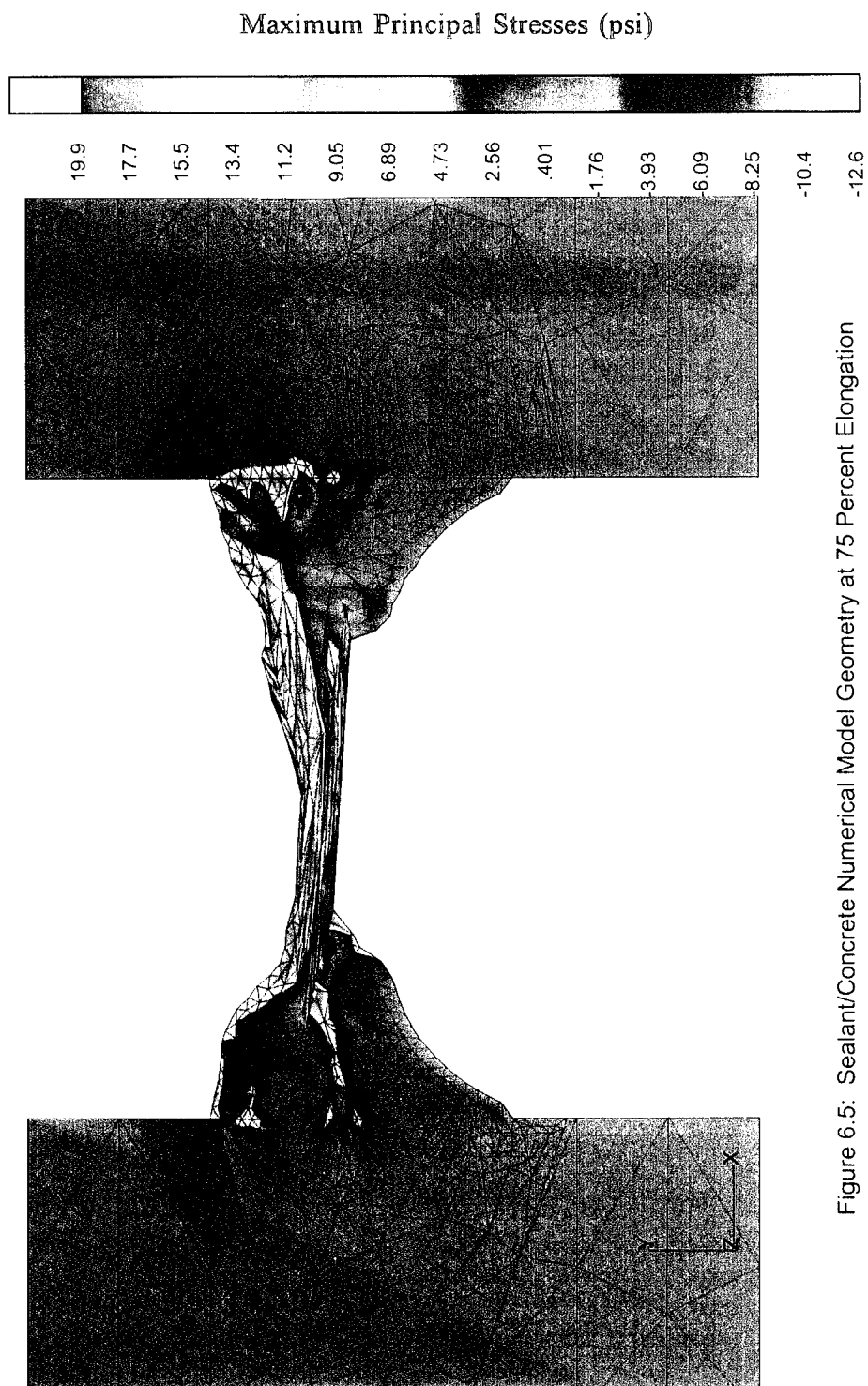
Figure 6.5: Sealant/Concrete Numerical Model Geometry at 75 Percent Elongation

There were a several areas of interest concerning the field simulation model. These areas of interest included the stress comparisons between the laboratory modeled results and the field modeled results and comparisons between the stress concentrations between the three and six node models.

As previously mentioned, the force per unit length for the 25 percent elongation was very similar to the force per unit length calculated at the higher displacement rates. The similarities were expected because the results of the DSR testing. The DSR test results indicated only small changes with changes in test temperatures. From time-temperature superposition, one would expect that the changes in shear modulus would also be small with changes in rates of loading.

The stresses that would develop in the sealant during movement induced by pavement faulting were also investigated using model. This movement was in the vertical direction and would occur at a high rate of displacement. Only one type of model was used in this portion of the investigation. The model consisted of only a vertical displacement of 0.25 inch (6.35 mm). No horizontal displacement was included in the model. Figure 6.8 illustrates the calculated maximum principal stresses for a vertical displacement of 0.25 inch (6.35 mm) within one second. The highest maximum principal stress is 24.6 psi (169.5 Pa) located at the top right sealant/concrete interface. This would be expected because there would be a peeling action of the sealant away from the concrete. Figure 6.9 illustrates the calculated principal stresses immediately after the concrete platen had returned to the original position. In this figure, the highest value of the maximum principal stresses had reduced to 3.66 psi (25.2 Pa). The results illustrated in Figures 6.8 and 6.9 demonstrated that during the rapid vertical loading, the silicone material behaved in an elastic manner as would be expected.

Stress Concentrations in the Three Node Versus Six Node Model

Another item of interest concerned the stresses illustrated in Figures 6.6 and 6.7 particularly at the interface between the concrete and sealant. The stress concentrations along this boundary appeared to be numerical artifacts from the

analysis and not realistic concentrations. This was verified by analyzing the six node model. The six node model exhibited stress concentrations at the top sealant/concrete interface and at the top of the parabolic surface on the bottom of the sealant. From a materials perspective this appeared more logical. Additionally, adhesive failures observed in the field were typically initiated at the top sealant/concrete interface. Therefore, it was concluded that the three node model could be used to calculate the force per unit length accurately but the six node model would need to be used to determine the stress concentrations in the sealant and along the sealant/concrete interface. However, even in the three node model, one of the highest areas of stress concentration was located at the top right edge of the sealant/concrete interface.

One of the controversial issues related to construction practices has been joint cleanliness. By combining DSR characterization of sealant materials with numerical modeling and laboratory tensile testing of sealant samples which have been prepared with a known amount of debris on the concrete blocks, one could quantify the cleanliness required to obtain a certain level of adhesion.

Maximum Principal Stresses (psi)

17.6 16.0 14.5 12.9 11.3 9.79 8.24 6.68 5.13 3.57 2.02 .461 -1.09 -2.65 -4.20 -5.76
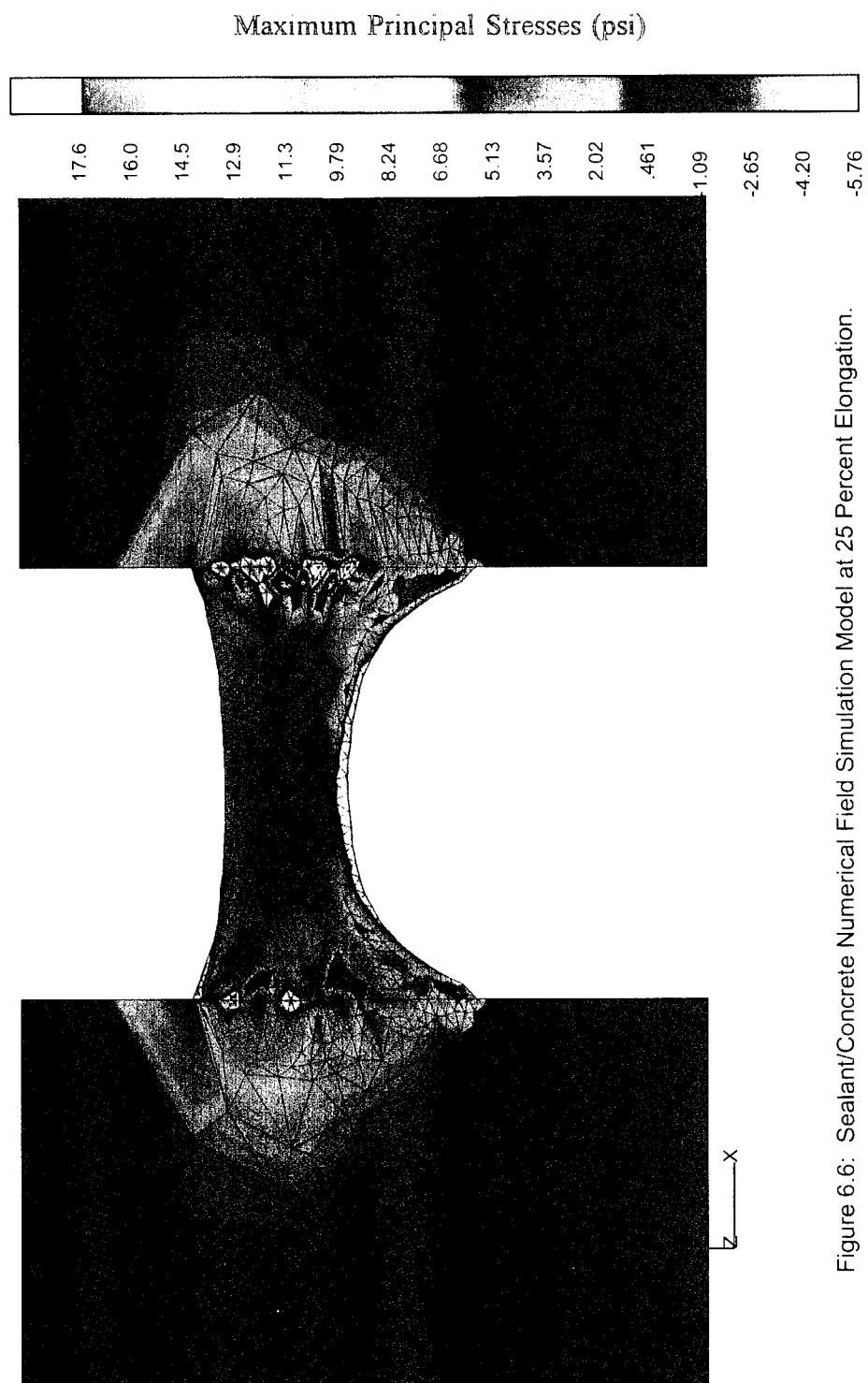
Figure 6.6: Sealant/Concrete Numerical Field Simulation Model at 25 Percent Elongation.

139

Maximum Principal Stresses (psi)

17.6 16.0 14.5 12.9 11.3 9.79 8.24 6.68 5.13 3.57 2.02 .461 -1.09 -2.65 -4.20 -5.76
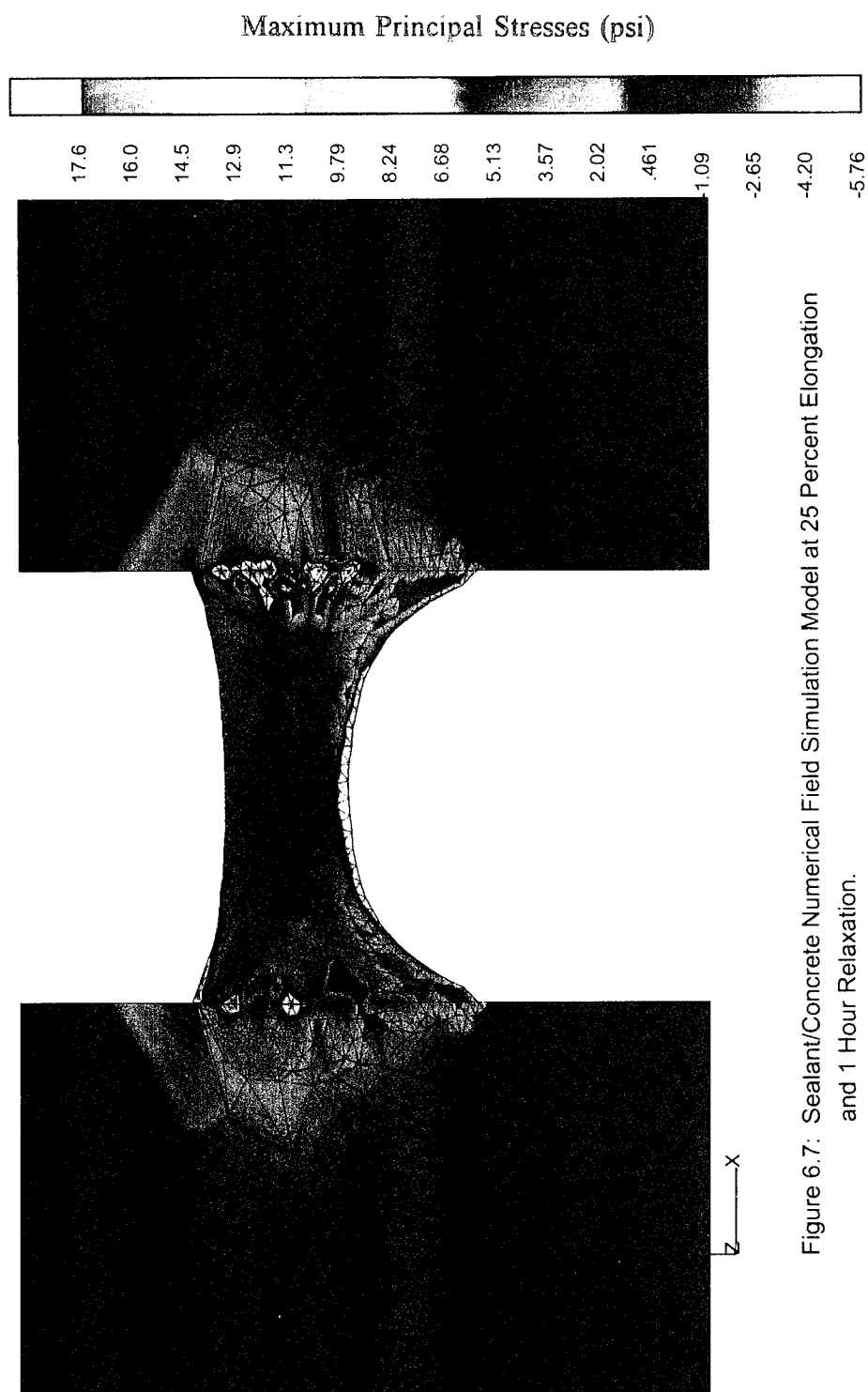
Figure 6.7: Sealant/Concrete Numerical Field Simulation Model at 25 Percent Elongation and 1 Hour Relaxation.

Maximum Principal Stresses (psi)

24.6 21.9 19.1 16.3 13.6 10.8 8.07 5.31 2.55 .206 -2.96 -5.72 -8.48 -11.2 -14.0 -16.7
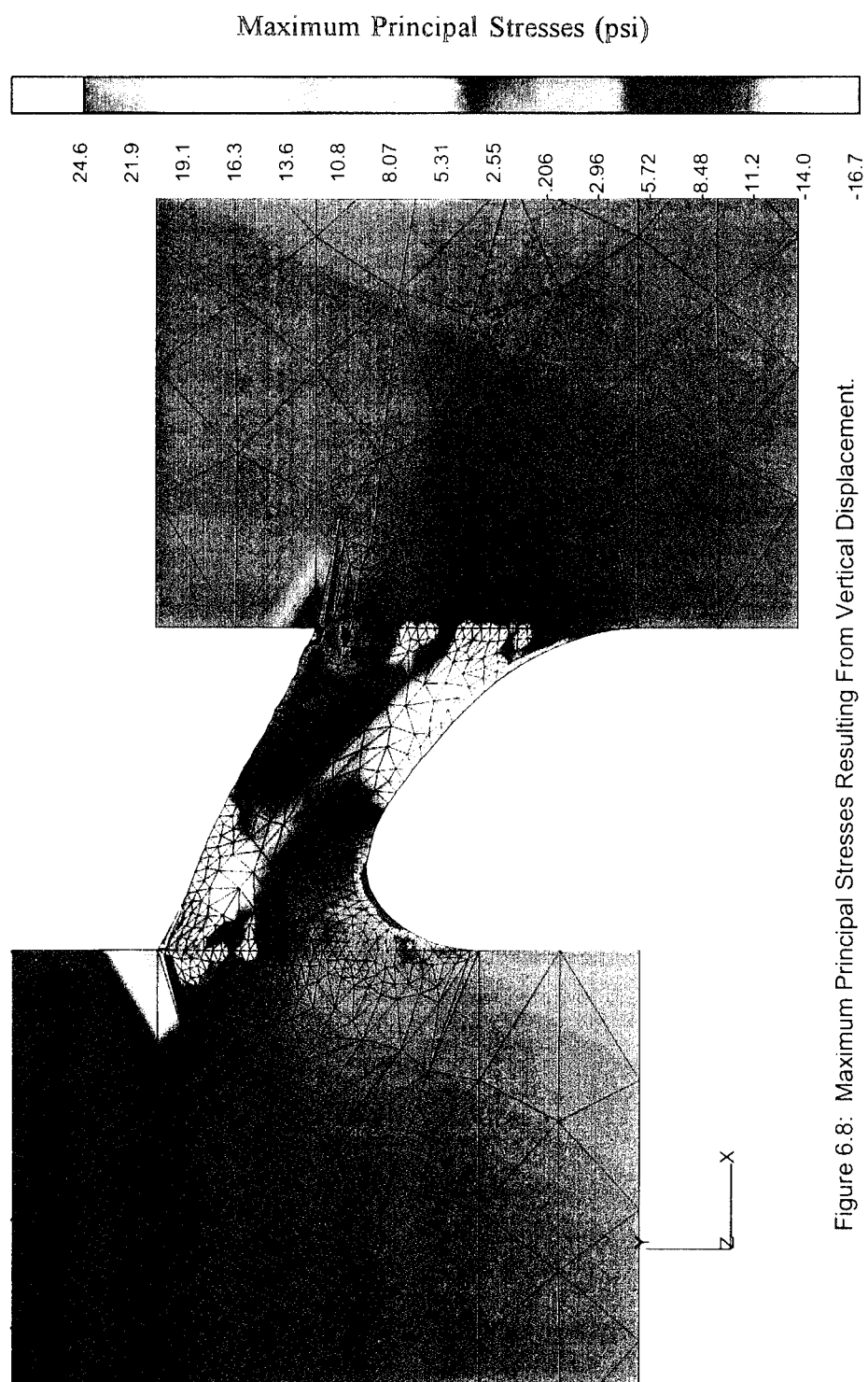
Figure 6.8: Maximum Principal Stresses Resulting From Vertical Displacement.

Maximum Principal Stresses (psi)

3.66	
3.21	
2.77	
2.32	
1.87	
1.43	
.979	
.532	
.0857	
-.361	
-.808	
-1.25	
-1.70	
-2.15	
-2.59	
-3.04	
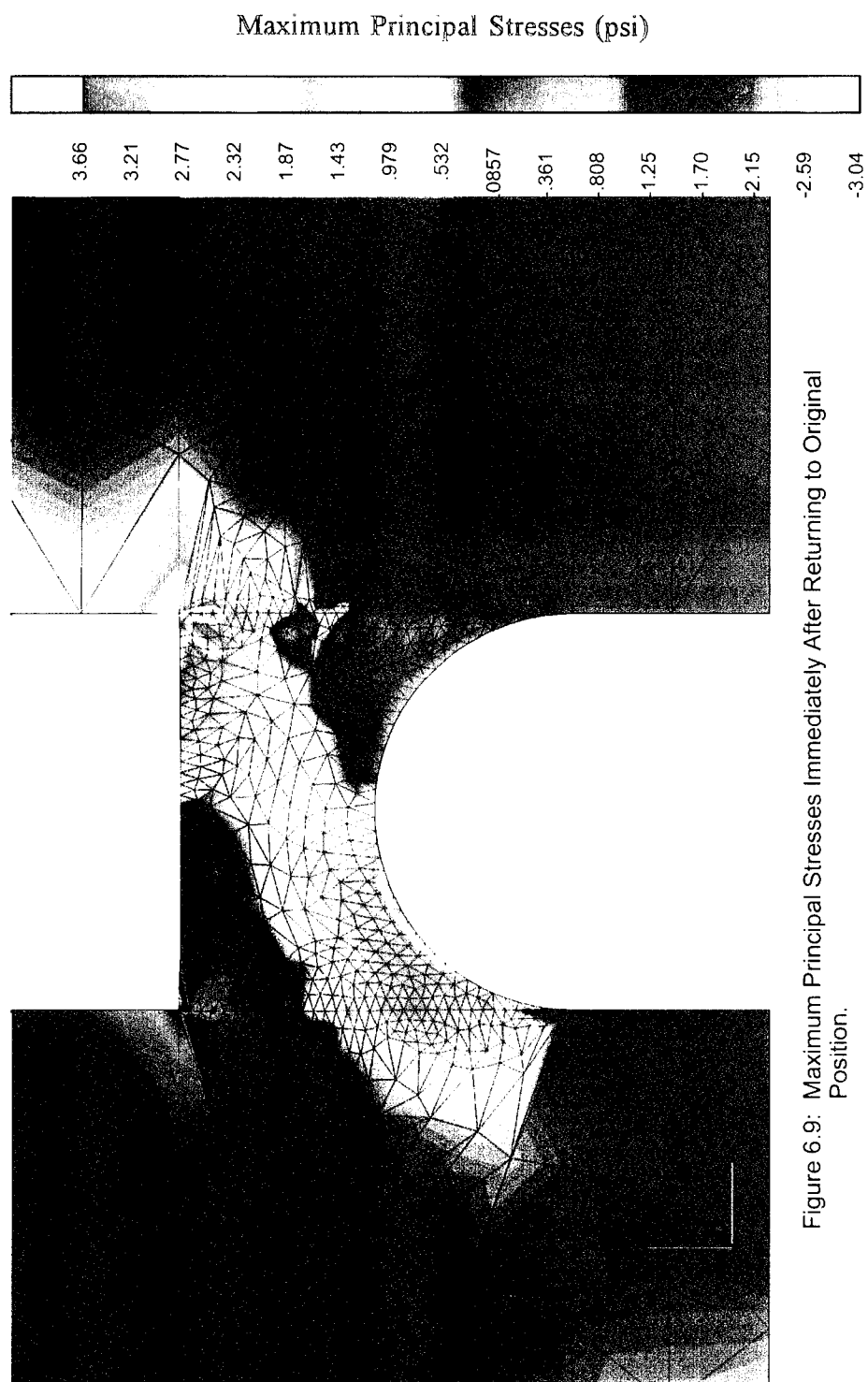
Figure 6.9: Maximum Principal Stresses Immediately After Returning to Original Position.

CHAPTER 7 - CONCLUSIONS AND RECOMMENDATIONS

The overall objective of this investigation was to develop an analytical methodology that would characterize silicone pavement joint sealants using DSR techniques coupled with numerical analysis. Additionally, it was expected that the DSR methodology could potentially be incorporated into existing specifications or be used as the basis for a new sealant specification. This overall objective was successfully met; the DSR characterization technique combined with numerical analysis provided information that was more directly related to field performance than the currently existing specifications. However, additional research will be required before the technique can be used as the basis for a new specification that supersedes current practice.

Conclusions

This research investigation verified many existing ideas concerning joint sealant materials as well as producing new insight. The specific conclusions of this research included:

1. Material Variability - The material variability of pavement joint sealants was very high. The variability of joint sealant materials even within the same lot number of material had been suspected for a number of years. Much of this variability was attributed to the fact that most sealants were produced from asphalt cements and coal tars, both of which were extremely variable in nature. Silicone sealants were believed to be less variable because they were manufactured from polydimethylsiloxane, a true polymer. The dynamic shear rheometer testing indicated that the variability of the silicone materials was also high. This variability could most likely be attributed to the fillers or some other additive in the sealant.

2. Material Characterization - The average material properties determined through dynamic shear rheometer and laboratory tensile testing appeared to be representative of the "true" material properties for elongations of up to 25 percent. The force per unit length at 25 percent elongation calculated by the three node numerical model using the DSR data input was within 0.4 percent of the values obtained from laboratory tensile testing. The force per unit length calculated by the numerical model at 50 percent elongation was less "accurate" than those calculated

at 25 percent elongation. The calculated force per unit length at 50 percent was within 18 percent of the laboratory obtained results. The potential cause of the deviation could be that the linear viscoelastic region of the material was exceeded at elongations of approximately 50 percent or more likely that the numerical model needed additional refinement for the very large displacements. Additionally, several samples (at least eight samples for silicone dynamic shear rheometer testing) must be tested to obtain the average value. This accuracy was verified through the numerical analysis at 25 percent elongation. The six node model exhibited a force per unit length that was approximately 12 percent lower than the laboratory determined results. However, the stress concentrations calculated with the six node model were more representative of those that would be expected from field observations. Therefore, the six node model should be used in the determination of stresses located at the sealant/concrete interface.

3. Material Specifications - The currently existing material specifications for pavement joint sealant materials did not directly relate to field performance. The dynamic shear rheometer testing could be related to field performance as demonstrated by the numerical analysis and laboratory tensile testing. However, conducting several tests on multiple samples to develop a discrete stress relaxation spectra for numerical analysis would not be feasible for most user agencies. Instead, two test temperatures should be selected for dynamic shear rheometer testing based upon the maximum and minimum in-use temperature the sealant would be exposed to for a given application. Criteria should then be developed to ensure that the sealant will perform as desired in that application. Criteria could not be established in this investigation because all of the materials had exhibited at least limited satisfactory performance in the field. Therefore, the failure envelope could not be determined because test results for materials that did not perform satisfactorily in the field were not obtained.

4. Accelerated Curing - All attempts to accelerate the curing of the silicone sealants were unsuccessful. The addition of water to the materials created

voids in the test specimens and the elevated temperature/humidity resulted in one material not curing. Therefore, for the purpose of specification requirements, the curing conditions used for the silicone should remain 21 to 28 days at laboratory conditions.

5. Accelerated Aging - Attempts to age the silicone samples using the pressure aging vessel were unsuccessful. The samples became saturated with air during the conditioning and upon release of the pressure, numerous voids formed in the test specimens. The forced-draft oven aging technique commonly used in current material specifications did apparently cause some aging in the sealant materials as demonstrated through the dynamic shear rheometer testing.

6. Model Verification - It was imperative that the model used to predict the behavior of a joint sealant material be verified through laboratory testing and that any extrapolation of the model beyond the regions that had been verified be conducted with extreme care. The comparison of the calculated forces per unit length at 25 and 50 percent versus the laboratory tensile data was an example of the potential problem of extrapolation. It was also extremely important that a mesh sensitivity analysis be conducted to ensure the results being obtained were not simply a result of the mesh that had been generated.

<div align="center">Recommendations</div>

This research project delineated several areas that require additional investigation or research. The specific recommendations of this research included:

1. Number of Samples - The average test results for the silicone sealants appeared to be representative of the material properties when a large number of samples were tested. The exact number of samples required for testing to obtain a representative characterization of all sealant materials needed to be determined. A total of eight samples were tested for the 21 day laboratory cured samples of sealant F. This was sufficient for the numerical modeling of laboratory tensile testing, but the statistical significance between various test temperature differences could not be ascertained.

2. Accelerated Curing - Two techniques were investigated and neither one proved completely successful. The addition of water to the sealants to accelerate the curing process created voids in the prepared samples. The voids adversely affected the determination of the material properties. Increasing the humidity around a sample using a small amount of water in a heated container prevented one of the materials from curing. A technique could not be used in a material specification if it automatically precluded the use of a material that had exhibited successful field performance. Accelerated curing is still a desirable goal for user agencies that are attempting to ensure they are receiving a high quality material. This goal can be achieved through two potential processes; develop an accelerated curing technique that would effectively cure all materials, or find an effective manner to implement quality product lists.

3. Numerical Analysis - The dynamic shear rheometer testing indicated that the materials did appear to age differently. The aging characteristics could be evaluated using the methodology described in this research; however, it would be useful to include the aging characteristics in the numerical analysis based on a specific climate of interest. To complete this effort, research would be required to delineate aging characteristics versus climate and expand the numerical model so that as time passed, the material properties of the sealant changed. Refinements to the numerical model to account for large displacements will also be required.

4. Adhesion Failures - This research indicated that the sealants could be adequately characterized using dynamic shear rheometry and numerical analysis. This analysis also allowed the stress concentrations in the sealant to be determined. One of the controversies associated with joint sealant projects has been the cleanliness of the joint before the sealant was installed. Research is required to quantify the cleanliness in the joint and provide guidance to user agencies concerning cleanliness of the joint versus reduced adhesive strength of the sealant. ABAQUS has special interface elements that could be used define the adhesive strength of the sealant, but research will be required to quantify the changes in the adhesive strength

of a sealant versus amount of debris on a joint face. This effort would require additional laboratory testing and refinements to the numerical model.

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Appendix - A

Silicone Pavement Joint

Sealant Material Specifications

(Specifications provided by ASTM D4.33,

Task Group 7 Chairman, Mr. Jim Chehovits)

Federal Aviation Administration

Silicone Join Sealant

1. DESCRIPTION

1.1 This item shall consist of providing and installing a silicone sealant capable of sealing joints in concrete pavements.

2. MATERIALS

2.1 Sealant. Silicone sealant (non-acid curing) material shall meet the requirements shown in Table 1.

Table 1. Silicone Sealant Requirements

Test Method	Test	Requirement
MIL-S-8802	Flow	0.3 maximum
MIL-S-8802	Tack free time	90 minutes maximum
ASTM D2240	Durometer hardness[1]	10 - 25
ASTM D412 (Die C)	Modulus at 150% elongation[1]	75 psi maximum
ASTM D412 (Die C)	Elongation[1]	800% minimum
MIL-S-8802	Adhesion to concrete	20 lbs. minimum
ASTM C719	Movement	± 50% minimum

[1] Sample cured 7 days at 77°F (25°C ± 2°C) and 50% ± 5% relative humidity.

Each lot or batch of sealing material shall be delivered to the jobsite in the manufacturer's original sealed container. Each container shall be labeled to include the following:

    a. Name of material

    b. Manufacturer's name

    c. Manufacturer's lot number

    d. Shelf life

    e. Mixing instructions

    f. Storage instructions

NAVAL FACILITIES ENGINEERING COMMAND

2.1.1.4 Single Component Cold-Applied Silicone

Silicone sealant shall be self-leveling, non-acid curing, and meet the following requirements:

Test	Test Method	Requirements
Weight Loss	ASTM C792 Modified[1]	10% maximum
Flow	ASTM C639 (Type I)	Smooth and level
Extrusion Rate	ASTM 603	30 seconds maximum
Tack Free Time	ASTM C679	5 hours maximum
Hardness (Shore 00)[2]	ASTM C661	30 - 60
Tensile Stress at 150% Elongation[2]	ASTM D412 (Die C)	30 psi maximum
Percent Elongation[2]	ASTM D412 (Die C)	700 minimum
Accelerated Weathering	ASTM C793	Pass 5000 hours
Bond and Movement Capability	ASTM C719	Pass 10 cycles at + 50% movement (no adhesion or cohesion failure)
Flame Resistance	FS SS-S-200	Pass

[1] Percent weight loss of wet (uncured) sample after placing in forced-draft oven maintained at 70°C ± 2°C for 2 hours.

[2] Specimen cured 21 days at 23°C ± 2°C and 50% ± 5% humidity.

ACCELERATED WEATHERING FACTORY TEST REPORT. For the accelerated weathering test, in lieu of testing the actual joint sealant to be used on the project, a report of a factory test, performed within two years of contract award, may be submitted.

STATE OF ARIZONA

Silicone Joint Sealant: is revised to read

Testing:

Silicone joint sealant shall be a low modulus silicone that is specifically formulated to seal Portland Cement Concrete joints. Silicone sealant shall be furnished in a one part formulation which is non-acid curing and shall meet the following physical requirements:

Test Method	Test	Material Requirement
ASTM D412 (Method A, Die C)	Tensile stress at 150% elongation[1]	45 psi maximum
ASTM D412 (Method A, Die C)	Elongation[1]	700% minimum
ASTM C603	Extrusion rate (ASTM C920, Type S, Grade NS)	25 seconds maximum
ASTM D792 (Method A)	Specific gravity	1.15 - 1.615
ASTM C679	Tack free time	120 minutes maximum
ASTM D2240 (Shore A)	Durometer hardness	25 maximum
ASTM C719 (mortar block)	Movement[2,3]	+50% and -50% of the joint width. Adhesive loss: maximum 15% of surface area and no cohesive failure after 10 cycles at standard conditions.

[1] Sample cured 7 days at 77°F ± 2°F and 50% ± 5% relative humidity.

[2] Curing of specimens shall be in accordance with Article 7.2; any option or alternate conditions will not be permitted,

[3] Section 8. Procedure, shall be strictly adhered to through and including Article 8.5. The test procedure will then be considered concluded.

STATE OF IOWA

B. Silicone joint sealer shall be a one-part silicone formulation intended for installation in highway pavement joints. The sealer shall cure to form a flexible, low modulus, high elongation silicone joint seal. A primer shall be used in the application when recommended by the manufacturer.

Backer rope used in conjunction with this sealer shall be of a closed-cell polyethylene, and no bond or reaction shall occur between the backer rope and sealer. The rope shall be of a size that compression is required for installation in the joint so it maintains its position during the sealing operation. Backer rope shall be dry.

1. Specific Requirements. The silicone joint sealer shall cure to a tack-free-to-touch condition at 77°F in less than 90 minutes. When cured for 14 days at a temperature of 74 to 80°F and 45 to 55% relative humidity, the silicone sealer shall meet the following test requirements:

Durometer hardness, Shore A	10 - 30
Tensile stress, 150% elongation, maximum	90 psi
Bond at -20°F, 3 cycles, 200% extension	No adhesive or cohesive failure
Accelerated weathering, 3,000 hours	No blisters or cracks

2. Method of Test. Testing shall be in accordance with the following methods:

Durometer hardness	ASTM D2240
Tensile stress	ASTM D412 (Die C)
Accelerated weathering	ASTM C793-75

STATE OF IOWA

(continued)

Bond : Prepare 1" x 1" x 3" concrete blocks in accordance with ASTM C719. A sawed face shall be used for the bond surface. Seal 2 inches of the block leaving 1/2 inch on each end of the specimen unsealed. The depth of the sealant shall be 1/2 inch and the width 1/2 inch. Cure the bond specimen in air for 14 days. Subject the sealer specimen to movement in accordance with ASTM C719 at a rate of 1/8 inch per hour. One cycle is 200% extension (to 1 1/2 inch width) and return to initial 1/2 inch width.

3. Acceptance. Inspection and acceptance of silicone joint sealer shall be in accordance with IM 436.02.

STATE OF COLORADO

Reference Specification Section 02515, Page 1, Part 2.01 A. First line after the word "shall" add the following "be non-acid cure and" Beginning with the fourth line add "in addition, the material shall meet the requirements shown in Table 1 below.

Table 1. Silicone Sealant Requirements.

Test Method	Test	Requirements
MIL-S-8802	Flow	0.3 maximum
MIL-S-8802	Tack free time	90 minutes maximum
ASTM D2240	Durometer hardness	10 - 25
ASTM D412 (Die C)	Modulus at 150% elongation[1]	75 psi maximum
ASTM D412 (Die C)	Elongation[1]	800% minimum
MIL-S-8802	Adhesion to concrete	20 lbs. minimum
ASTM C719	Movement	± 50% minimum

[1] Sample cured 7 days at 77°F ± 2°F and 50% ± relative humidity.

STATE OF GEORGIA

1. Physical Requirements:

Type Silicone	A	B	C
Tensile stress at 150% strain[1] (maximum psi)	45	40	15
Durometer hardness, Shore[1] (0°F and 77°F ± 3°F	"A" 10 - 25	"00" 40 - 80	"00" 20 - 80
Bond to concrete mortar[1,3] (minimum psi)	50	40	35
Tack free time[2] (skin-over), maximum minutes	90	90	90
Extrusion rate (minimum grams/minute)	75	90	100
Non-volatile (minimum %)	90	90	90
Shelf life from date of shipment	6 months	6 months	6 months
Movement capability and adhesion[1]	No adhesive or cohesive failure after 10 cycles at 0°F		
Ozone and U.V. resistance[1]	No chalking, cracking, or bond loss after 5,000 hours		

[1] The cure time for these specimens shall be 21 days for Type A and 28 days for Types B and C. Specimens shall be cured at 77°F ± 3°F and 50% ± 5% relative humidity.

[2] At conditions of 77°F ± 3°F and 50% ± 5% relative humidity.

[3] Type C silicone must also meet its bond strength requirement to asphalt concrete.

STATE OF NORTH CAROLINA

928-4 LOW MODULUS SILICONE SEALANT

Low modulus silicone sealant shall be furnished in a one part silicone formulation. A primer for bond to concrete shall be used when required in accordance with the manufacturer's recommendations.

The sealant shall meet the following requirements:

Flow	0.3 inches maximum
Extrusion rate	75-250 grams/ minute
Tack free time at 77°F ± 3°F and 45 - 50% relative humidity	20 - 75 minutes
Specific gravity	1.01 - 1.515
Durometer hardness, Shore A, cured 7 days at 77°F ± 3°F and 45 - 50% relative humidity	10 - 25
Tensile stress at 150% elongation, cured 7 days at 77°F ± 3°F and 45 - 50% relative humidity	75 psi maximum
Elongation, cured 7 days at 77°F ± 3°F and 45 - 50% relative humidity	500% minimum
Peel (adhesion); unprimed aluminum substrate with aluminum screen, cured 7 days at 77°F ± 3°F at 45 - 50% relative humidity	20 lbs minimum at least 75% cohesive failure

Tests shall be performed in accordance with the following test methods:

Flow	MIL-S-8802
Extrusion rate	MIL-S-8802
Tack free time	MIL-S-8802
Specific gravity	ASTM D792, Method A
Durometer hardness	ASTM D2240
Tensile stress	ASTM D412 (Die C)
Elongation	ASTM D412 (Die C)
Peel	MIL-S-8802

STATE OF KANSAS

SUBSECTION 1507 SILICONE JOINT SEALANT

1507.01 DESCRIPTION

This specification cover silicone joint sealant and backer rod to be used for filling joints in Portland cement concrete pavement.

1507.02 REQUIREMENTS

(a) Joint Sealant - Silicone joint sealant shall be prequalified prior to use on Department projects. The joint sealant shall be either Type I (non self-leveling) or Type II (self-leveling). Joint sealants shall be a one-part, cold-applied silicone formulation which is self priming to and compatible with Portland cement concrete. Acetic acid cure sealants are not acceptable. furthermore, Type II sealants shall be easy to place in concrete pavement joints, shall be self-leveling within the joint and shall provide a satisfactory surface configuration without tooling. The silicone sealants shall comply with the following applicable requirements:

PROPERTY	REQUIREMENTS	
	Type I	Type II
Skin development time, minutes	120 maximum	120 maximum
Cure-through	pass	pass
Extrusion rate, grams/minute	90 - 250	200 - 600
Non-volatile content, %	90 minimum	90 minimum
Bond to concrete at 0°F, 5 cycles, 100% extension	pass	pass
Compression set	pass	pass
Elongation, %	600 minimum	1,000 minimum

(b) Backer Rod - The material furnished for this purpose shall be resilient closed or open cell polyethylene foam rod as recommended by the manufacturer of the sealant. It shall be compatible with the silicone sealant and no bond or reaction shall occur between the rod and sealant.

STATE OF TEXAS

Class 5. (Low Modulus Silicone Sealant for Concrete Pavement Joints). The material shall be furnished in a one-part silicone formulation which does not require a primer for bond to concrete. A backer rod shall be required which shall be compatible with the sealant. No bond or reaction shall occur between the rod and sealant.

The material shall meet the requirements of Table I.

TABLE I. PHYSICAL PROPERTIES

Property	Requirement	Test Procedure
Flow, inches	0.2 maximum	MIL-S-8802D, Section 4.8.4
Extrusion rate, grams/minute	90 - 250	MIL-S-8802D, Section 4.8.5
Tack free time, 77 ± 2°F, minutes	35 - 75	MIL-S-8802D, Section 4.8.7
Durometer hardness, Shore A	10 minimum	ASTM D2240
Adhesive Strength[1] 　Initial, 7 day cure, 77 ± 2°F, psi 　After water immersion, psi 　After heat aging, psi 　After cycling at 0°F	 10 - 50 10 - 50 10 - 50 10 - 50	 Tex-525-C Tex-525-C Tex-525-C Tex-525-C

[1] There shall be no evidence of crack, separation or other opening that at any point is over 1/8 inch deep in the sealer or between the sealer and test blocks.

Class 6 (Self-leveling, Low Modulus Silicone Sealant for Asphalt and Concrete Pavements Joints). This shall be a single component, self-leveling silicone material that is compatible with both asphalt and concrete pavement. The sealer shall not require a primer for bond. A backer rod shall be required which shall be compatible with the sealant. No reaction shall occur between the rod and sealant.

The physical properties of the self-leveling sealant shall meet the following requirements:

STATE OF TEXAS

(concluded)

Property	Requirements	Test Procedure
Tack free time, 77 ± 2°F, minutes	120 maximum	Tex-525-C
non-volatile content, %	93 minimum	Tex-525-C
Adhesive Strength[1] Initial, 10 day cure, 77 ± 2°F, psi	4 minimum	Tex-525-C
After water immersion, psi	4 - 30	Tex-525-C
After heat aging, psi	4 - 30	Tex-525-C
After cycling at -20°F	4 - 30	Tex-525-C

[1] After the specimens have been cured and subjected to water immersion, heat aging and cycling, they shall be subjected to tensile loading at the rate of 0.5 ± 0.05 inch per minute until the joint material has been extended 150 percent. The load at 150 percent shall be recorded and 1-1/4 inch spacers inserted to maintain the extension for 24 hours. After 24 hours, the specimens shall be examined for adhesive or cohesive failure.

Appendix - B

Proposed Test Method

for Silicone Pavement Joint Sealants

Standard Method of Testing Single-Component Silicone Joint Sealants

for Concrete Pavements

(DRAFT)

1. Scope

    1.1   This method covers the test method for rheologically evaluating single-component silicone joint sealant materials used to seal joints and cracks in concrete pavements. This test method provides an indication of the temperature susceptibility of the sealant material and allows low and high temperature stiffness to be evaluated. In addition, the data obtained using this test procedure can be used as a basis for finite element analysis of the sealant in a joint or crack.

    1.2   This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. Some guidance concerning safety and health issues can be obtained by reviewing the sealant manufacturer's Material Safety Data Sheet (MSDS) and precautionary statements from the testing apparatus manufacturer.

2. Significance and Use

    2.1   The method outlines procedures for determining viscoelastic material properties of single-component silicone joint sealants. The joint sealant, when inserted in the joint must adhere to the pavement substrate and remain flexible throughout it's life cycle to ensure that water infiltration through the joint and debris retention in the joint is minimized. The viscoelastic material properties obtained using this procedure provide a measurement which relate to the ability of the sealant to remain flexible during it's life cycle by comparing unaged properties to accelerated aged properties. The flexibility of the sealant is determined by specifically measuring the modulus of the sealant material at different temperatures and different rates of loading. The results obtained from this procedure can be used to develop a discrete

relaxation spectra for the sealant which can then be used in numerical modeling to compare sealant performance. The procedures outlined herein do not address the ability of the sealant to adhere to the pavement substrate.

3. Equipment

3.1   Requirements - The following equipment will be required to perform the test procedures specified herein:

3.1.1   Controlled Strain Dynamic Shear Rheometer - A controlled strain dynamic shear rheometer capable of applying the specified strain ± 0.025 percent and maintaining a normal force on the sample as specified ± 0.1 gram.

3.1.2   Environmental Test Chamber - The environmental chamber shall be compatible with the dynamic shear rheometer such that it can maintain the temperature of the test specimen to within ± 1.0°C of the specified temperature during testing.

3.1.3   Sample Dispenser - The sealant material shall be dispensed into the sample mold using either a pneumatic or hand operated caulking gun.

3.1.4   Forced Draft Oven - capable of maintaining a temperature of 93.3 ± 1°C (200 ± 2°F).

4. Sampling

4. Sampling - Unless otherwise agreed to by the manufacturer or contractor and the user agency, the samples for testing shall be taken at the point of manufacturer. It shall be the responsibility of the contractor to determine that the samples taken are representative of the batches of material to be used on a specific project. The representative composite sample shall consist of not less than 3.78 L (1 gal) of material.

5. Conditioning

5. Conditioning - Laboratory Atmospheric Conditions, hereafter referred to as standard laboratory conditions, are 23 ± 2°C (73 ± 4°F) and 50 ± 5 percent relative

humidity. Specimens shall be cured and stored at standard laboratory conditions prior to testing unless otherwise specified.

6. Specimen Preparation - Prepare duplicate samples in molds with inside dimensions of 75 mm by 75 mm by 1.5 mm (3 in by 3 in by 0.06 in). The molds may be constructed as one piece or two pieces. The molds shall be coated with a release agent. The molds shall be overfilled with the sealant material and all excess shall be struck off leaving a smooth, void free, specimen. The specimens shall be allowed to cure in the laboratory at standard laboratory conditions for 21 days ± 4 hours. At the end of the 21 days, one specimen shall be tested as specified below and the second specimen shall be placed in a forced-draft oven set at 93.3 ± 1°C (200 ± 2°F) for 7 days ± 4 hours. At the end of the conditioning period the sample shall be tested as specified below.

7. Specimen Testing - After the material has been cured for 21 days and conditioned in the forced-draft oven, if required, remove the sealant from the mold and punch two samples 25 mm diameter specimen disks from the cured material. Place one of the specimens between the parallel plates of the dynamic shear rheometer and adjust the gap opening so that the normal force reading on the sample is 50 ± 10 newtons. Close the DSR environmental chamber around the test specimen and set the test temperature at -30 ± 1.0°C. Readjust the gap opening so that the normal force is set to 50 ± 10 newtons and allow the sample temperature to equilibrate for 5 minutes ± 30 seconds. Programmed the dynamic shear rheometer to create sinusoidal deformation at frequencies of 0.02, 0.04, 0.06, 0.08, 0.1, 0.2, 0.4, 0.6, 0.8, 1.0, 2.0, 4.0, 6.0, 8.0, and 10.0 Hz. The frequency test sequence shall be repeated so that four measurements are taken at each frequency using an amplitude of 15 percent. After testing is completed at the -30°C, the test temperature is increased by 10°C, the gap is adjusted so that the normal force reading on the sample is 50 ± 10 newtons, the sample temperature is allowed to equilibrate, and the sample is

tested using the above frequencies. The process is continued until all test temperatures from -30 to 50°C in 10°C increments was completed. The first sample is removed from the parallel plate configuration, the second sample is inserted and the test sequence is repeated. This testing sequence is then repeated with the 7 day, forced-draft oven aged samples.

8. Reporting - The results shall be reported in graphical form of complex modulus (G^*) versus frequency or G^* versus temperature.

Appendix - C

Typical Physical Properties

of the Silicone Sealants

as Provided in the Manufacturer's Literature

Table C1. Typical Physical Properties of Sealant A (Obtained from Manufacturer's Literature)

Uncured Property	Test Specification	Result
Density at 25°C	not provided in literature	1.34 g/cm^3
Consistency	DIN 52 454, profile B	non-slump
Extrusion Rate	3 mm nozzle, pressure = 0.21 N/mm^2, temperature = 23°C	4 ml/10 sec.
Skin Forming Time	23°C and 50% relative humidity, sample size 18 mm deep and 24 mm diameter	30 - 60 min.
Shrinkage at Cure	DIN 52 451	6%
Curing Rate	73°C and 50% relative humidty measured in dishes of 18 mm deep and 24 mm diameter 7 Days 14 Days 21 Days 28 Days	5.0 mm deep 7.0 mm deep 9.0 mm deep 11.0 mm deep

Cured Property	Test Specification	Result
Tensile Strength	DIN 53 504-S 3 A DIN 52 455-01	1.1 N/mm^2 0.45 N/mm^2
Elongation at Break	DIN 53 504-S 3 A DIN 52 455-01	800% 400%
Modulus of Elongation	DIN 53 504 at 100% elongation DIN 52 455-01 at 25% elongation DIN 52 455-01 at 100% elongation BS 5889 (1980 clause 8) at 75% elongation	0.30 N/mm^2 0.15 N/mm^2 0.25 N/mm^2 0.23 N/mm^2
Shore A Hardness	DIN 53 505	16
Tear Strength	ASTM D 624, Die B	5.0 N/mm^2
Density at 25°C	DIN 53 479	1.37 g/cm^3

Table C2. Typical Physical Properties of Sealant B (Obtained from Manufacturer's Literature)

Uncured Property	Test Specification	Result
Extrusion Rate	MIL-S-8802	250 gm/min
Tack-Free Time	ASTM C 679	81 minutes
Flow	ASTM D 2202	0.0 mm
Curing Rate	23°C and 50% relative humidity measured on a sample that is 12.7 mm by 12.7 mm 5 Days	12.7 mm deep

Cured Property	Test Specification	Result
Elongation to Failure	ASTM D 412, Die C	958 percent
Modulus at 150% Elongation	ASTM D 412, Die C	38.9 psi
Shore A Hardness	ASTM D 2240	11
Specific Gravity	ASTM D 792-A	1.24
Bond and Movement Capability	ASTM C 719 (± 50%)	Pass 10 Cycles
Accelerated Weathering	ASTM C 793	Pass 5,000 Hours
Bond to Mortar	AASHTO T-132	81 psi
Tensile Adhesion (elongation to failure)	ASTM D 3583, Section 14 (modified)	606 percent

Table C3. Typical Physical Properties of Sealant C (Obtained from Manufacturer's Literature)

Uncured Property	Test Specification	Result
Extrusion Rate	MIL-S-8802	90 to 250 gm/min
Tack-Free Time	MIL-S-8802	60 minutes
Flow	MIL-S-8802	0.0 mm
Through Cure	23°C and 50% relative humidity measured on a sample that is 12.7 mm by 12.7 mm 7 to 14 Days	12.7 mm deep

Cured Property	Test Specification	Result
Elongation to Failure	ASTM D 412, Die C	1200 percent minimum
Modulus at 150% Elongation	ASTM D 412, Die C	45 psi
Shore A Hardness	ASTM D 2240	15
Specific Gravity	ASTM D 792-A	1.45 to 1.515
Bond and Movement Capability	ASTM C 719 (+ 100%/-50%)	Pass 10 Cycles
Accelerated Weathering	ASTM C 793	Pass 5,000 Hours
Tensile Strength at Maximum Elongation	Not provided in the literature.	100 psi
Tensile Adhesion (elongation to failure)	ASTM D 3583, Section 14 (modified)	500 percent minimum

Table C4. Typical Physical Properties of Sealant D (Obtained from Manufacturer's Literature)

Uncured Property	Test Specification	Result
Extrusion Rate	ASTM C 603	145 gm/min
Tack-Free Time	ASTM C 679	71 minutes
Flow	ASTM D 2202	2.54 mm
Through Cure	23°C and 50% relative humidity measured on a sample that is 6.35 mm 7 to 14 Days	6.35 mm deep

Cured Property	Test Specification	Result
Elongation to Failure	ASTM D 412, Die C	700 percent
Modulus at 150% Elongation	ASTM D 412, Die C	43 psi
Shore A Hardness	ASTM D 2240	15
Specific Gravity	ASTM D 792-A	1.18
Bond and Movement Capability	ASTM C 719 (±50%)	Pass 10 Cycles
Tensile Strength at Maximum Elongation	ASTM D 412	90 psi

Table C5. Typical Physical Properties of Sealant E (Obtained from Manufacturer's Literature)

Uncured Property	Test Specification	Result
Extrusion Rate	MIL-S-8802	580 gm/min
Skinover Time	Georgia DOT-106	45 minutes
Leveling at 77°F	ASTM C 639	Pass
Curing Rate	23°C and 50% relative humidity measured on a sample that is 12.7 mm by 12.7 mm 14 Days	12.7 mm deep

Cured Property	Test Specification	Result
Elongation to Failure	ASTM D 412, Die C	884 percent
Modulus at 150% Elongation	ASTM D 412, Die C	22.1 psi
Shore 00 Hardness	ASTM D 2240	64
Specific Gravity	ASTM D 792-A	1.29
Bond and Movement Capability	ASTM C 719 (± 50%)	Pass 10 Cycles
Accelerated Weathering	ASTM C 793	Pass 5,000 Hours
Bond to Mortar	AASHTO T-132	61.1 psi
Tensile Adhesion (elongation to failure)	ASTM D 3583, Section 14 (modified)	694 percent

Table C6. Typical Physical Properties of Sealant F (Obtained from Manufacturer's Literature)

Uncured Property	Test Specification	Result
Extrusion Rate	Not provided in the literature.	400 gm/min
Skinover Time	Not provided in the literature.	30 minutes
Through Cure	23°C and 50% relative humidity	14 days

Cured Property	Test Specification	Result
Elongation to Failure	ASTM D 412, Die C	1600 percent
Modulus at 150% Elongation	ASTM D 412, Die C	10 psi
Shore 00 Hardness	ASTM D 2240	40
Specific Gravity	ASTM D 792-A	1.3 to 1.4
Accelerated Weathering	ASTM C 793	Pass 5,000 Hours
Tensile Adhesion (elongation to failure)	ASTM D 3583, Section 14 (modified)	600 percent minimum

Appendix - D

Rheological Data of Joint Sealant Materials

Table D1. Sealant A Storage Modulus Data at 1Hz After Curing.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.0	33,667	18,261	68,500	12,243	79,500	12,598	93,500	837
-19.9	37,667	14,976	92,500	30,125	83,833	10,420	101,000	9,859
-9.8	43,167	12,238	88,333	23,738	84,000	5,138	103,500	18,076
0.2	60,667	7,312	83,667	17,896	84,000	0	99,167	15,943
10.4	53,667	817	82,500	16,979	80,167	983	96,000	15,336
20.2	59,500	7,868	77,500	15,527	75,500	548	94,167	17,348
30.2	58,500	7,868	74,000	14,241	72,333	516	89,000	16,125
40.0	56,333	6,593	72,167	12,968	69,167	408	85,167	15,158
49.9	54,333	6,593	54,333	6,593	66,500	548	83,167	12,271

[1] Avg = Average
[2] SD = Standard Deviation

Table D2. Sealant A Storage Modulus Data at 1Hz After Curing and 7 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.2	100,000	10,955	92,000	8,050	74,000	39,436	103,667	6,976
-19.9	125,000	5,477	110,000	0	79,000	44,913	120,000	0
-9.6	125,000	5,477	115,000	16,432	80,333	43,454	130,000	10,955
0.3	115,000	5,477	130,000	21,909	74,500	38,888	130,000	10,955
10.4	110,000	0	130,000	10,955	75,000	38,341	110,000	0
20.2	105,000	5,477	125,000	16,432	68,500	34,507	110,000	6,325
30.0	98,500	1,643	115,000	16,432	67,167	33,048	101,167	9,683
40.0	94,833	1,722	109167	11,873	64,167	30,857	98,667	12,453
49.8	90,500	2,429	107,000	14,241	61,167	29,762	N/A[3]	N/A

[1] Avg = Average
[2] SD = Standard Deviation
[3] Data Not Available

Table D3. Sealant A Storage Modulus Data at 1Hz After Curing and 14 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.0	115,000	5,477	65,667	32,867	86,833	10,068	81,833	7,521
-19.7	113,167	18,444	72,167	30,492	97,167	14,063	109,833	29,620
-9.7	130,000	10,955	72,667	27,023	97,500	13,693	101,000	20,814
0.2	120,000	10,955	65,500	32,316	90,500	10,407	93,167	18,444
10.2	125,000	16,432	70,000	23,004	94,667	16,801	87,500	13,693
20.4	115,000	16,432	53,667	15,706	86,333	13,156	84,167	14,063
30.0	110,000	10,955	55,000	16,432	85,167	16,253	80,167	14,428
40.0	107,667	13,515	53,667	16,071	82,000	15,336	76,667	12,801
50.0	100,500	10,407	52,667	14,611	78,167	14,798	71,833	12,238

[1] Avg = Average
[2] SD = Standard Deviation

Table D4. Sealant A Storage Modulus Data at 1Hz After 21 Days Laboratory Curing.

Average Temperature (°C)	Test 1		Test 2		Test 3	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.4	72,500	24,304	93,500	837	111,667	4,083
-19.6	80,667	32,135	101,000	9,859	130,000	10,955
-9.8	77,167	28,896	103,500	18,076	130,000	10,955
0.4	80,000	32,863	99,167	15,943	125,000	5,477
10.1	80,000	32,863	96,000	15,336	125,000	5,477
20.4	74,500	27,934	94,167	17,348	118,333	9,832
30.1	73,667	27,023	89,000	16,125	111,667	4,083
40.1	70,667	23,738	85,167	15,158	110,000	0
49.9	67,833	22,825	83,167	12,271	100,000	0

[1] Avg = Average
[2] SD = Standard Deviation

181

Table D5. Sealant B Storage Modulus Data at 1Hz After Curing.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-29.8	26,500	1,643	31,000	2,191	23,667	817	80,833	3,125
-19.7	25,500	1,643	31,500	2,739	25,500	3,834	78,500	6,025
-9.6	26,000	2,191	32,167	3,125	26,000	4,382	79,500	1,643
0.2	25,500	2,739	31,667	3,670	26,000	4,382	76,000	2,191
10.4	25,500	2,739	31,000	3,286	25,667	5,125	80,500	6,025
20.5	23,000	2,191	28,667	3,670	24,500	3,834	77,167	5,672
29.9	22,500	1,643	26,667	2,944	23,500	3,834	47,500	1,643
40.1	21,000	1,095	24,500	3,834	22,500	2,739	45,167	983
50.0	19,500	548	23,000	3,286	22,000	2,191	43,000	0

[1] Avg = Average
[2] SD = Standard Deviation

Table D6. Sealant B Storage Modulus Data at 1Hz After Curing and 7 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.0	25,000	4,382	N/A[3]	N/A	31,500	548	93,500	4,930
-19.3	30,167	3,869	N/A	N/A	24,500	8,216	95,667	4,761
-10.2	35,500	4,930	N/A	N/A	N/A	N/A	94,000	6,229
0.2	36,000	1,095	N/A	N/A	24,500	9,311	89,000	6,573
10.4	37,000	1,095	N/A	N/A	30,667	1,862	84,833	8,232
20.3	35,500	1,643	46,500	2,739	29,833	2,041	80,500	8,216
29.8	34,500	1,643	45,667	516	N/A	N/A	N/A	N/A
40.2	33,000	2,191	42,667	516	N/A	N/A	N/A	N/A
50.1	31,500	1,643	40,500	548	N/A	N/A	N/A	N/A

[1] Avg = Average
[2] SD = Standard Deviation
[3] Data Not Available

Table D7. Sealant B Storage Modulus Data at 1Hz After Curing and 14 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.0	62,500	23,552	N/A[3]	N/A	23,000	3,286	71,333	9,136
-19.7	73,000	4,382	N/A	N/A	32,500	12,598	71,000	6,573
-9.6	67,833	5,672	N/A	N/A	22,500	548	69,833	5,672
0.2	65,000	4,382	N/A	N/A	23,000	0	71,500	548
10.2	63,833	4,215	N/A	N/A	31,167	8,954	71,000	1,095
20.2	59,667	3,670	N/A	N/A	29,833	8,232	68,167	1,329
30.2	56,167	3,125	N/A	N/A	30,000	8,764	66,000	0
40.1	53,833	2,401	N/A	N/A	28,500	8,216	63,833	408
49.9	51,000	3,286	N/A	N/A	27,500	7,120	61,500	548

[1] Avg = Average
[2] SD = Standard Deviation
[3] Data not available

184

Table D8. Sealant C Storage Modulus Data at 1Hz After Curing.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.1	62,667	8,824	58,833	11,514	73,000	40,532	256,667	160,707
-19.4	59,833	7,494	70,000	20,455	69,167	33,778	255,000	125,976
-9.8	61,667	7,686	78,000	24,100	70,000	32,502	258,333	96,833
0.3	64,833	9,326	90,000	32,863	70,500	29,029	248,333	74,944
10.4	N/A[3]	N/A	94,833	27,571	70,833	25,380	285,000	16,432
20.3	67,500	9,311	95,000	27,386	68,667	21,182	223,333	69,474
30.3	66,333	8,406	97,167	25,015	68,333	16,071	210,000	65,727
40.2	65,000	7,668	94,000	17,527	66,167	12,254	198,333	64,005
50.1	65,333	8,406	95,833	15,523	66,000	9,879	190,000	54,772

[1] Avg = Average
[2] SD = Standard Deviation
[3] Data not available

185

Table D9. Sealant C Storage Modulus Data at 1Hz After Curing and 7 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-29.8	44,000	26,291	38,833	1,329	N/A[3]	N/A	104,333	50,031
-19.7	52,333	29,944	46,833	3,869	N/A	N/A	102,500	44,881
-9.7	16,600	12,488	54,167	7,885	46,500	9,311	125,000	16,432
0.4	22,100	17,418	60,167	14,442	47,000	6,573	125,000	16,432
10.3	22,100	17,418	69,667	19,367	48,500	3,834	120,000	21,909
20.3	55,833	17,714	71,667	19,005	46,000	3,286	116,500	18,802
30.2	53,500	14,789	67,833	21,367	44,500	2,739	92,000	30,679
40.1	52,500	12,243	65,167	20,999	42,500	2,739	95,167	27,206
50.1	50,667	10,231	64,667	19,725	41,000	2,191	96,167	26,126

1 Avg = Average
2 SD = Standard Deviation
3 Data not available

187

Table D10. Sealant C Storage Modulus Data at 1Hz After Curing and 14 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.2	22,500	548	40,500	3,834	49,167	8,976	104,333	17,178
-19.2	32,500	2,074	49,500	6,775	71,333	4,412	120,000	21,909
-9.6	43,500	4,930	61,833	25,748	95,167	16,253	125,000	16,432
0.4	50,333	7,312	71,500	22,828	117,667	46,375	125,000	16,432
10.5	67,333	20,087	58,000	0[3]	107,667	24,468	125,000	16,432
20.4	73,000	24,836	80,667	21,182	101,500	20,266	118,333	13,292
30.3	73,000	24,100	79,500	22,457	93,167	18,444	115,000	16,432
40.1	70,500	20,266	78,333	21,191	92,500	19,170	115,000	16,432
50.1	69,500	20,266	74,333	18,991	86,500	14,789	114,333	17,166

[1] Avg = Average
[2] SD = Standard Deviation
[3] Only one sample tested

Table D11. Sealant D Storage Modulus Data at 1Hz After Curing.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing[3]	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.1	47,667	5,854	48,333	5,854	47,667	9,136	19,667	577
-20.0	52,000	2,191	50,500	7,120	54,167	10,048	27,000	0
-9.8	55,333	3,011	49,500	7,120	54,333	10,231	31,667	577
0.4	60,167	10,048	46,833	6,401	48,667	6,947	35,000	0
10.2	61,333	13,515	45,167	5,307	55,333	14,611	35,000	0
20.5	55,500	10,407	43,333	5,854	47,500	6,411	33,000	0
30.1	52,000	9,859	40,500	6,025	47,000	5,477	31,000	0
39.9	48,500	8,216	40,000	6,573	43,667	4,761	29,667	577
50.1	45,167	7,859	39,000	7,668	43,000	4,382	27,000	0

[1] Avg = Average
[2] SD = Standard Deviation
[3] Only one sample tested.

189

Table D12. Sealant D Storage Modulus Data at 1Hz After Curing and 7 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-29.9	57,167	4,579	100,000	0	69,833	4,215	40,333	18,991
-19.3	59,000	6,229	105,000	5,477	78,833	6,047	43,833	12,238
-9.4	58,167	7,494	125,000	5,477	90,167	21,729	45,000	9,859
0.5	60,500	11,572	120,000	0	76,500	6,025	47,833	8,232
10.7	59,667	12,420	110,000	0	76,000	5,865	52,667	2,582
20.2	56,167	11,143	110,000	0	72,667	5,854	53,333	3,670
30.3	54,333	11,326	99,833	408	70,667	7,312	52,667	1,506
40.2	52,333	10,231	87,167	3,764	69,333	7,312	51,167	983
50.0	50,000	8,764	81,667	1,211	65,167	7,859	50,167	983

[1] Avg = Average
[2] SD = Standard Deviation

Table D13. Sealant D Storage Modulus Data at 1Hz After Curing and 14 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.3	61,667	30,690	64,000	2,191	95,167	5,307	17,000	5,477
-19.8	77,667	21,556	74,333	577	110,000	10,955	21,000	2,967
-9.6	89,000	23,013	70,500	3,507	108,167	12,968	24,500	548
0.4	77,667	11,378	91,333	20,452	101,667	9,136	27,000	1,095
10.8	77,167	4,957	95,833	26,476	52,667	2,582	24,500	10,407
20.4	79,833	983	89,333	22,642	108,333	4,083	25,000	9,859
30.2	69,167	7,494	85,333	23,653	97,833	2,483	24,500	9,311
40.2	71,500	3,886	80,333	21,547	101,167	9,725	24,667	9,136
50.2	71,833	5,707	77,000	18,995	90,833	7,139	23,667	8,406

[1] Avg = Average
[2] SD = Standard Deviation

Table D14. Sealant E Storage Modulus Data.[1]

Average Temperature (°C)	After 21Days Laboratory Curing		After 21Days Laboratory Curing and 7 days Oven Aging		After 21Days Laboratory Curing and 14 days Oven Aging	
	Avg[2] G' (Pa)	SD[3] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.1	19,667	2,944	22,500	2,739	17,833	5,307
-19.6	21,500	4,930	26,500	3,834	20,500	8,216
-9.4	22,000	5,477	27,000	4,382	21,500	8,216
0.5	23,500	6,025	26,500	4,930	21,500	8,216
10.4	24,000	6,573	27,000	3,286	22,500	7,120
20.5	23,500	6,025	26,167	3,488	22,000	6,573
30.3	22,667	5,854	26,334	2,582	21,167	6,765
40.1	22,000	5,477	25,500	2,739	20,167	6,765
50.1	21,500	6,025	25,000	2,191	20,000	6,573

[1] The samples which were exposed to the accelerated curing conditions did not cure; therefore, they were not tested.
[2] Avg = Average
[3] SD = Standard Deviation

Table D15. Sealant F Storage Modulus Data at 1Hz After Curing.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-29.3	53,333	7,312	52,500	2,739	46,333	8,406	41,667	16,071
-19.6	52,500	6,025	47,333	2,582	49,333	817	47,500	15,884
-9.6	49,500	3,834	46,167	5,672	48,167	408	51,500	11,502
0.2	N/A[3]	N/A	46,500	7,120	44,000	3,286	55,500	4,930
10.2	38,167	8,954	N/A	N/A	44,333	817	58,333	516
20.3	39,500	7,120	46,667	9,501	42,500	548	59,500	4,930
30.3	39,000	5,477	45,500	10,407	41,000	1,095	57,833	5,672
40.2	38,500	3,834	45,500	8,216	39,500	548	56,000	6,573
50.1	37,500	2,739	43,500	8,216	36,000	2,191	54,667	7,312

[1] Avg = Average
[2] SD = Standard Deviation
[3] Data not available

Table D16. Sealant F Storage Modulus Data at 1Hz After Curing and 7 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
30.0	66,167	4,215	52,667	6,593	44,000	10,955	29,500	7,120
-19.7	60,000	4,382	52,000	4,382	45,167	14,063	34,000	12,049
-9.7	55,667	4,033	51,500	548	41,500	12,598	36,500	13,693
0.2	57,000	1,095	48,000	3,286	39,000	10,955	39,500	14,789
10.3	54,500	548	47,666	3,670	38,000	10,955	40,000	15,336
20.3	52,000	0	31,400	22,242	36,000	9,859	38,833	14,428
30.2	50,000	0	45,000	1,095	35,500	9,311	38,000	14,241
40.1	48,000	0	43,833	1,329	34,000	8,764	37,500	12,598
50.0	46,000	0	42,500	1,643	33,000	8,764	36,833	12,238

[1] Avg = Average
[2] SD = Standard Deviation

194

Table D17. Sealant F Storage Modulus Data at 1Hz After Curing and 14 Days Oven Aging.

Average Temperature (°C)	After 24 Hours Accelerated Curing		After 48 Hours Accelerated Curing		After 72 Hours Accelerated Curing		After 21 Days Laboratory Curing	
	Avg[1] G' (Pa)	SD[2] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30.1	34,333	6,947	42,500	6,025	38,500	10,407	53,833	20,634
-19.9	46,667	4,761	54,667	1,862	41,333	9,136	55,500	18,075
-9.7	45,667	3,670	47,500	7,120	42,833	8,589	55,500	15,884
0.2	44,167	2,401	50,500	1,643	45,167	6,765	54,500	14,789
10.4	43,000	2,191	48,500	2,739	45,000	5,477	55,000	13,145
20.3	42,000	2,191	46,000	3,286	44,000	4,382	53,500	12,598
30.2	50,000	5,477	44,000	3,286	43,500	2,739	52,500	12,598
40.1	48,500	4,930	30,033	14,967	43,000	2,191	51,500	11,502
49.9	44,833	3,125	29,233	14,441	42,000	2,191	50,833	11,873

[1] Avg = Average
[2] SD = Standard Deviation

Table D18. DSR Verification Testing Results.

Average Temperature (°C)	Sealant C 21 Day Laboratory Cure[1]		Sealant F 24 Hour Accelerated Cure[4]		Sealant F 24 Hour Accelerated Cure, 24 Hour Pressure Aged[5]	
	Avg[2] G' (Pa)	SD[3] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30	32,833	6,873	42,667	11,381	47,667	15,939
-20	43,083	7,354	40,722	11,896	46,167	15,302
-10	53,917	13,070	39,500	12,157	42,667	15,529
0	61,083	16,323	40,111	12,083	40,750	14,536
10	64,083	16,632	36,889	10,571	39,500	13,284
20	64,417	16,903	36,667	9,917	37,083	12,154
30	61,917	15,871	35,833	9,288	37,583	10,022
40	59,917	14,700	35,278	8,477	36,167	9,504
50	57,750	14,220	35,556	7,801	35,167	9,024

1 Average of six samples, samples had voids and bubbles on one surface.
2 Avg = Average
3 SD = Standard Deviation
4 Average of six samples.
5 Average of four samples.

Table D19. Sealant F 24 Hour Accelerated Cured, Pressure Aged Test Results.

Average Temperature (°C)	Unaged[1]		24 Hour Pressure Aged[4]		48 Hour Pressure Aged[5]	
	Avg[2] G' (Pa)	SD[3] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30	42,667	11,381	47,667	15,939	45,667	9,836
-20	40,722	11,896	46,167	15,302	42,889	7,271
-10	39,500	12,157	42,667	15,529	43,000	7,246
0	40,111	12,083	40,750	14,536	40,667	6,144
10	36,889	10,571	39,500	13,284	44,833	3,869
20	36,667	9,917	37,083	12,154	43,500	2,739
30	35,833	9,288	37,583	10,022	42,000	2,191
40	35,278	8,477	36,167	9,504	40,000	2,191
50	35,556	7,801	35,167	9,024	38,667	2,944

[1] Average of six samples.
[2] Avg = Average
[3] SD = Standard Deviation
[4] Average of four samples.
[5] Average of three samples.

Table D20. Sealant F 72 Hour Accelerated Cured, Pressure Aged Test Results.[1]

Average Temperature (°C)	Unaged		24 Hour Pressure Aged		48 Hour Pressure Aged[5]	
	Avg[2] G' (Pa)	SD[3] G'	Avg G' (Pa)	SD G'	Avg G' (Pa)	SD G'
-30	33,500	3,834	39,500	1,643	44,333	2,582
-20	30,111	3,180	36,000	1,095	39,000	2,191
-10	34,000	0[4]	33,500	548	35,500	2,739
0	28,500	4,930	31,000	1,095	32,167	2,401
10	27,500	4,930	29,500	1,643	30,000	2,191
20	25,833	4,579	27,500	1,643	28,000	2,191
30	24,500	4,930	25,833	1,329	26,000	2,191
40	23,000	4,382	24,500	1,643	24,000	2,191
50	23,500	4,930	24,167	1,329	23,000	2,191

[1] All results are an average of two samples except as noted.
[2] Avg = Average
[3] SD = Standard Deviation
[4] Only one sample

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13. ABSTRACT *(Maximum 200 words)*

Joint sealant materials began to be widely used in pavement applications in an effort to protect the structural integrity of the pavement during the early 1940's. The protection was derived by minimizing moisture intrusion through the joint and preventing debris retention in the joint. Material specifications were soon developed to provide a means of material quality control. These specifications have evolved slowly over time, but they have not kept pace with the technological advancements made in the joint sealant industry, and, in some instances, the specifications have impeded technological advancements. Additionally, the properties measured in the specifications do not provide a direct correlation to field performance. Therefore, a need existed for a joint sealant characterization methodology that would provide a more direct correlation to field performance. The methodology should include the aging characteristics of the sealant and take into consideration the viscoelastic nature of the materials. A methodology was developed during this research that determines material properties related to field performance. Specifically, dynamic shear rheology was used to determine the viscoelastic response of six silicone pavement joint sealants. The storage and loss moduli versus frequency mastercurves were used to calculate a discrete stress relaxation spectrum which was converted into input for numerical analysis. The force per unit length modeled using this methodology was within 1 percent of the force per unit length measured in laboratory tensile testing at 25 percent elongation.

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These results indicate that the methodology developed using dynamic shear rheology combined with numerical analysis provides satisfactory characterization of the sealant material properties. The methodology can be used to assist in the selection of a sealant for a given application, to determine the cause of failures experienced in the field, and to determine the remaining life of sealants.